Tappi

PUBLISHED BY THE TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY

JANUARY 1952

PEXOL*

(HERCULES FORTIFIED SIZE)

AN IMPORTANT DEVELOPMENT FOR PAPER MAKERS

Hercules' Pexol, formerly known as Dresinate® 209, is gaining wide use among paper mills all over the country.

Why?

Because this fortified rosin size is cheaper to use than conventional sizes—mill operators report that Pexol is highly efficient, particularly for difficult sizing problems.

Pexol is readily available in paste or dry form. Try it and prove to yourself how this fortified rosin size can help you solve difficult sizing problems and save money, too.

Hercules' research is constantly at work to produce new and better products for the paper industry such as Pexol and Size-Aid. Illustrated above is the Hercules Photometer, a precision tool developed by Hercules to measure accurately transudation rates of liquids through paper.

For further information and technical data about Hercules' research in the papermaking industry, or for samples of Pexol or Size-Aid, write to:

SIZE-AID*

GAINS WIDE USE, TOO

Hercules' Size-Aid, formerly known as Dresinate® 211, is a dry, concentrated size additive formulated to increase the efficiency of rosin size.

HERCULES POWDER COMPANY
INCORPORATED
Paper Makers Chemical Dept., 967 King Street, Wilmington, Dela.

*TRADE-MARK

DESIGN DETAIL Sales and Engineering Departments cooperate to insure that each machine will meet the specific needs of the mill. Al Olson (*left*) and Dick Smith (*right*) check with C. E. Bieber, Chief Draftsman, as to placement of doctors. Personal attention to design detail is an essential ingredient in the building of every high-speed, high-production Beloit paper machine.—*Beloit Iron Works, Beloit, Wis.*

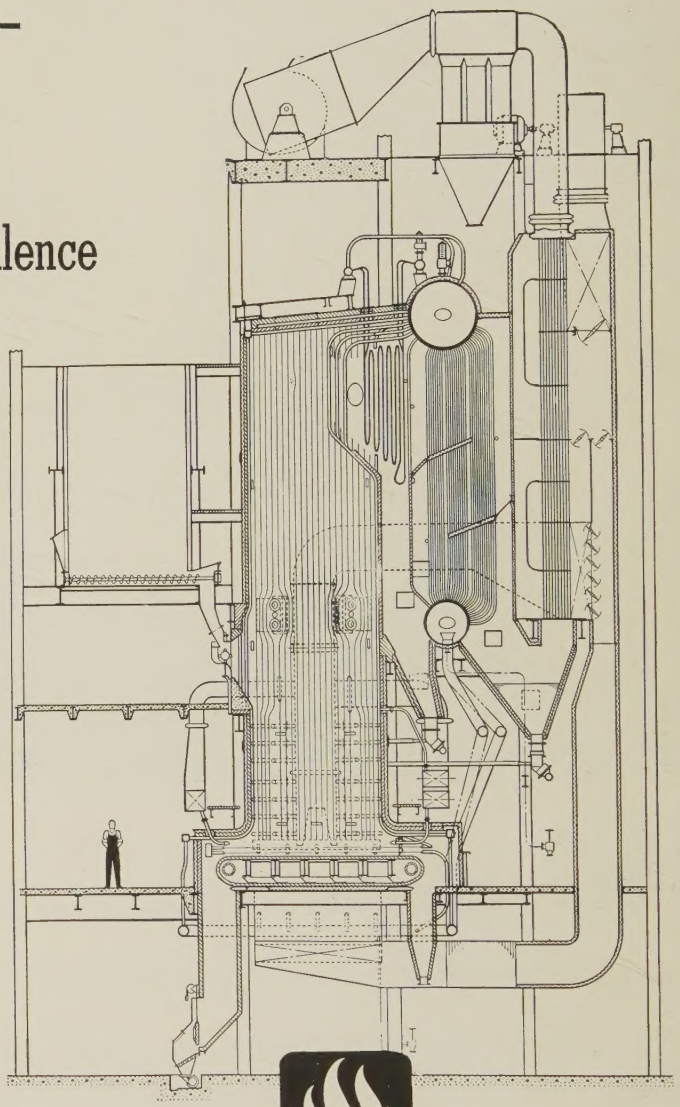
BELOIT

PAPER MACHINERY

WHEN YOU BUY BELOIT...YOU BUY MORE THAN A MACHINE!

New Method of Bark Burning

A revolutionary combination of—
Flash Drying—
Suspension Burning—
Extreme Turbulence



With the new C-E method of bark burning the bark spreader distributors are located in the front furnace wall at a considerable height above the grate surface. Combustion air is introduced through several rows of nozzles arranged so that air streams will impinge on each other tangentially. The direction of air rotation is reversed in each alternate row. The turbulent effect thus obtained fills the furnace with a swirling gas mass, that flash drys the bark and burns most of it in suspension. The rest is deposited in an even layer on the grate where it quickly burns to completion. The design, likewise, has proved highly successful for burning oil, alone, or in combination with the bark. It is equally adaptable to the use of gas as an alternate fuel.

In a paper presented before a recent Tappi meeting, the chief engineer of a large Southern paper company, discussed his installation of this equipment in detail. Of particular significance were these comments. "The savings in fuel cost in this installation represents a major portion of the financial return on invested capital. The reduction in direct operating labor resulting from the incorporation of the bark boiler into our main power plant building as well as the elimination of maintenance on refractory settings and water cooled grates has likewise effected an appreciable savings." In his report he also pointed out that performance has been excellent and availability exceptionally high — actually better than 99 per cent.

Here again a C-E development has provided the answer to a major fuel burning problem . . . a problem of widespread importance to the pulp and paper industry ie., a reliable and efficient means of burning bark and wood refuse. The services of the Combustion organization are freely available to your engineers and consultants. B-537

C-E

COMBUSTION ENGINEERING—SUPERHEATER, INC.

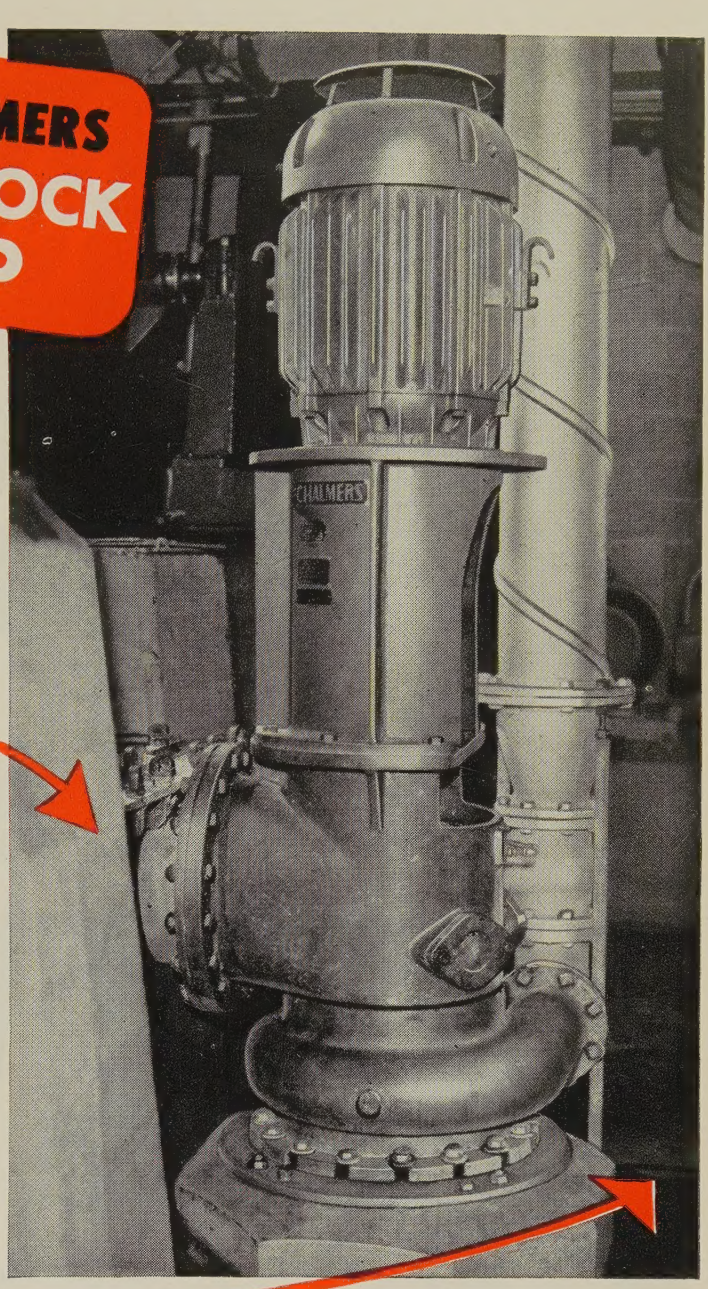
200 Madison Avenue, New York 16, N. Y.

PRODUCTS FOR THE PAPER INDUSTRY INCLUDE RECOVERY UNITS, STEAM GENERATING, FUEL BURNING AND RELATED EQUIPMENT; ALSO PRESSURE VESSELS

New ALLIS-CHALMERS PAPER STOCK PUMP

Vertical Design-

Saves Floor Space!



FLOOR SPACE COSTS MONEY and you can save a substantial amount by using the new Allis-Chalmers Vertical Paper Stock Pump. In new installations, it may be possible to tuck the pump away in a corner and thus utilize otherwise empty space. In modernization programs, a vertical pump may be the only method of fitting new equipment into existing space.

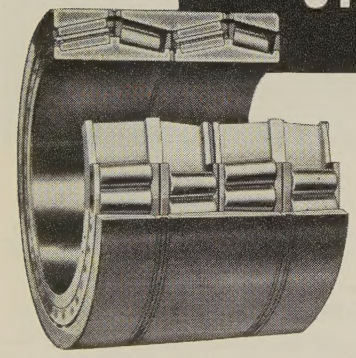
The basic pump design is the same as the reliable and performance-proved Type PW horizontal design. Notice the extremely large inlet which is standard regardless of pump size. Designed to handle up to 8% stock (when used with *Hi-Density* Feeder) and available in many materials, the Allis-Chalmers Vertical Paper Stock Pump is well suited to almost every paper mill operation. It will efficiently handle all types of acid and alkaline liquors as well as all types of high consistence stock.

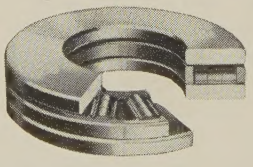
COMPLETE PUMPING UNIT

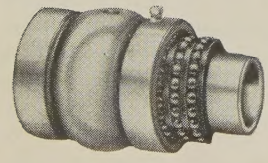
Allis-Chalmers can supply the complete pumping unit — pump, motor and control — all of one coordinated design and manufacture. You don't have to worry about matching components and divided responsibility. And Allis-Chalmers pump application engineers are familiar with all phases of paper mill operation. They know your problems and can help you solve them. Get complete information on the new Allis-Chalmers Vertical Paper Stock Pump and what it can do for you. Call your nearby Allis-Chalmers District Office or write Allis-Chalmers, Milwaukee 1, Wisconsin. A-3567

Hi-Density is an Allis-Chalmers trademark.

ALLIS-CHALMERS







Solving special friction problems is an everyday job with Torrington. Designing and producing made-to-order bearings has been an important part of our business for years.

This extensive experience has included all types of anti-friction bearings . . . both small and large . . . and for use in virtually every type of equipment. Result: Torrington can give you an impartial recommendation tailored to *your* application.

Whether you are a builder or user of machinery requiring bearings, Torrington engineers will gladly work with you in selecting a standard—or designing a special bearing tailored to your needs.

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South Bend 21, Ind. Torrington, Conn.

District Offices and Distributors in Principal Cities of United States and Canada

TORRINGTON BEARINGS

Spherical Roller • Tapered Roller • Straight Roller • Needle • Ball • Needle Rollers

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DORRCO DOINGS IN 1951

The year is ending with many changes of fundamental importance. England's new administration, though facing colossal problems, naturally makes believers in Free Enterprise more hopeful. Millions of us here today are thrilled by the hope that we can vote for a really great man, one like our first president, a great general, but above all an able and very wise patriot. No one believes that he would leave his present duties unless he felt that as president he could better serve his country and the world. It is inspiring to realize that he certainly can count on our strongest leaders to help build his team. A statement last year, that "America was in greater danger from within than without," is recalled when we think of the disclosure of lamentably lowered standards in many areas — a normal product, I would say, of stressing the doctrine that the State "owes" so much to all its citizens rather than that the citizen owes services to the State, if Democracy is to survive.

* * *

ABROAD — As here, our associates in Europe have had their greatest year. Among our projects starting this year were Colne Valley Sewage Plant, west of London, one of the largest sludge filtration and drying/incineration plant in the world, and a large metallurgical plant for Mines de Bor in Yugoslavia.

RELIEVING THE SULPHUR SHORTAGE — Sulphuric acid manufacturers, sulphite pulp producers and other users of sulphur dioxide, faced with a world-wide shortage of elemental sulphur, have found the Dorrco FluoSolids System an economically feasible means of utilizing iron sulphides as an alternate source of SO_2 gas. Using pyrite running 48-50 percent sulphur and yielding a gas averaging 14-15 percent SO_2, or pyrrhotite with lower sulphur content and yielding a slightly weaker gas, FluoSolids has brought both installation and operating costs down to a reasonable level. Concurrently, development work continues in such fields as the calcination of water softening and chemical pulp mill lime sludges; the sulphating roasting of copper concentrates prior to leaching and direct electrolytic precipitation without smelting; and the roasting of zinc flotation concentrates for either electrolytic, retort or zinc fuming operations.

WORLD'S LARGEST TRICKLING FILTERS—The revitalized sewage treatment plant of the City of Baltimore, Maryland, now has 30 acres of trickling filters, served by 50 Dorrco Sewage Distributors, each 157 ft. 6 in. in diameter and designed to handle a total flow of 112 million gallons per day. Our association with this new "world's largest" dates from 1930 when we sold the city its first Dorr Clarifiers which are still in service.

ION-EXCHANGE — Two large Dorrco De-ionization Systems are scheduled to start up about the end of the year, purifying organic chemical solutions. Regardless of the nature of specific applications, we supply a complete, integrated service for handling all kinds of ion-exchange projects from the initial analysis of the problem to the starting up of the new installation.

DORRCO AERATOR-CLARIFIER — The new combination of two well-known principles has demonstrated its worth at Idabel, Oklahoma, and elsewhere. It fills a real need wherever it is desired to increase present removals of suspended solids and BOD'S—wherever odor control is a necessity—or where flocculation, with or without chemicals, is beneficial. It is readily adaptable both to primary and secondary treatment.

INDUSTRIAL WASTES — Greater attention is being given to solutions of this problem as business executives realize good public relations require that their plants create no offense and furnish effluents comparable with those of domestic treatment systems. Significantly, many large companies have appointed engineers with the sole responsibility of caring for their plant effluents.

DORRCO VACUATOR — This versatile sewage pre-treatment unit has this year convincingly demonstrated its worth and flexibility. One installation in Oklahoma removes scum, grit and some suspended solids from raw feed. Another, in California, pre-treats various trade wastes ahead of the municipal treatment plant. Still another, in Maryland, acts as a grease removal device.

CONSULTING ENGINEERING—During the year, Dorr-designed plants for producing the first triple superphosphate in England and magnesia from sea water in Norway were put into operation. Design jobs abroad have included a copper leaching installation in the Near East, a triple superphosphate plant for Greece and a phosphoric acid plant for Switzerland. Projects in this country covered such diversified operations as recovering blast furnace flue dust, remodeling a large gold cyanide plant, and producing SO_2 gas for the manufacture of sulphite pulp.

INDUSTRIAL WATER — The largest single equipment order booked in our history, will be used for industrial water purification in an amount approximating that daily used in New York City.

* * *

The high point for me in 1951 was a quick month spent in Europe revisiting most of our Associates. It was my first trip since '48 and I found great improvement both in physical conditions and in the mental attitude of all we met. Most interesting was a three-day session in honor of the completion of our new office building at Herengracht 478, Amsterdam. Three hundred years old and full of the dignity and real beauty of an older period, when merchants both worked and lived in the same home, it has been remodelled for our needs with loving care. Our group, foregathered there, represented twelve nations.

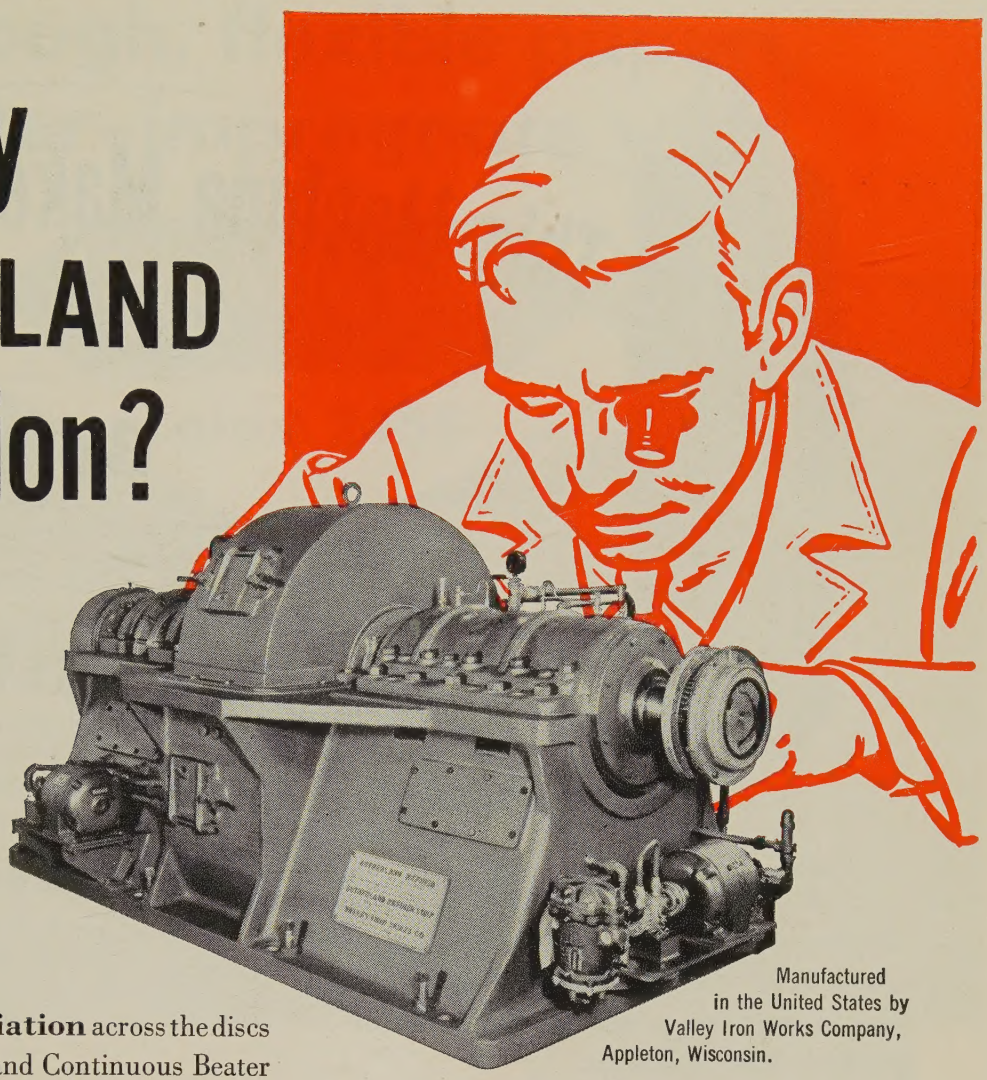
In Paris and Wiesbaden new and larger offices have been acquired and all our staff, crowded with work, bear testimony to their efficient organization and cooperation, and to the improved conditions in Europe.

It has been a busy year, with our organization keyed to meet the expansion inevitably brought about by world activity. We all join in sending New Year's Greetings to our Staff, Associates, and friends Worldwide, with the strong conviction that while "the impossible takes a little longer," it will be done, and that we can now see ahead, dimly perhaps, a picture of the World as we would wish it.

Barry Place, Stamford, Conn.

DORRCO

Why SUTHERLAND precision?



Manufactured in the United States by Valley Iron Works Company, Appleton, Wisconsin.

The maximum deviation across the discs of this 42-inch Sutherland Continuous Beater is only two thousandths of an inch. And in addition to this precision machining, all parts are assembled to minimum tolerances. Why is this eleven-ton machine made so carefully . . . how does this accuracy pay off in your mill?

Most important, it assures you of a degree of uniformity in stock preparation that is otherwise impossible to attain. By means of inlet pressure and power readings, your operators can quickly reset Sutherlands to get desired stock characteristics. Furthermore, these machines are built to give you this precision-control year after year. Records in Southern kraft mills show that total maintenance costs, including disc replacement after fifteen years, are *less than five cents per ton.*

Write today for a copy of "Modern Stock Preparation," and get the complete story on solid satisfaction with Sutherlands.

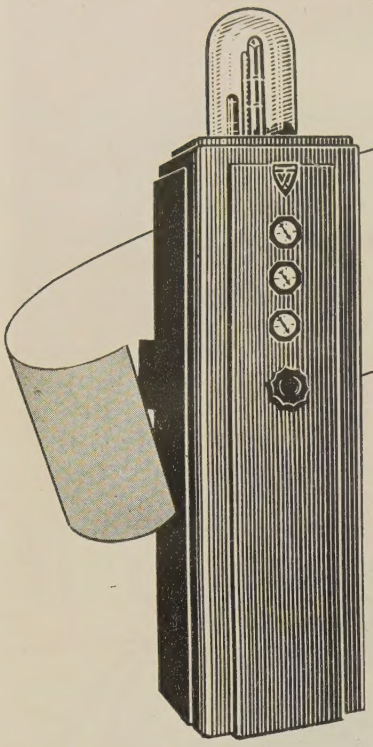
SUTHERLAND

continuous beating systems

Designed, Engineered, Serviced

***by* SUTHERLAND REFINER CORPORATION**

TRENTON 8, N. J.



This Machine Makes Paper

Better...

Faster...

Cheaper...

The machine is a W&T Visible Vacuum Chlorinator for the controlled and dependable application of chlorine to stock, white water and fresh water supply to destroy slime and odor producing micro-organisms.

It makes paper BETTER because by eliminating bacterial spots and off-odors it improves quality — obviously essential in food papers.

It makes paper FASTER because it reduces down-time due to sheet breakage, prevents the formation of slime in felts and improves water removal.

And, finally, it makes paper CHEAPER because it reduces stock, chemical and heat losses, improves grade and saves labor.

Write today to learn more about why leading paper and board mills everywhere have turned to W&T Chlorinators to solve bacteria-induced paper-making problems.

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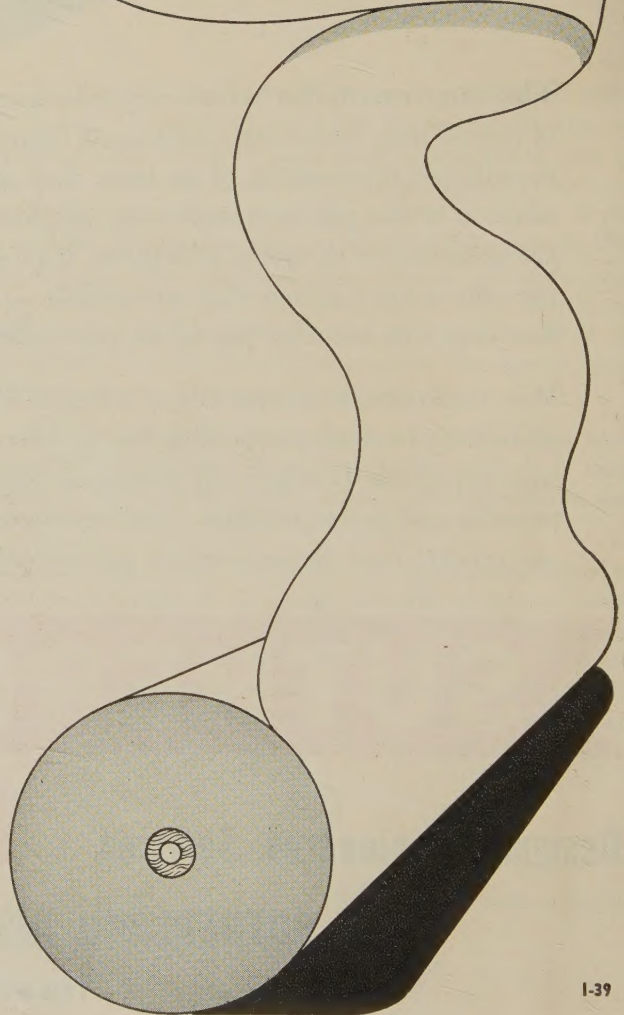
CHLORINE AND CHEMICAL CONTROL EQUIPMENT
NEWARK 1, NEW JERSEY • REPRESENTED IN PRINCIPAL CITIES



I-39

For tough, flexible papers, beater-impregnate with

CHEMIGUM Latex 245B

BEATER impregnation is possible with Chemigum latices—producing papers with high tear resistance and elongation. Inset: laboratory test shows resistance to edge tear of such papers.

CHEMIGUM LATEX 245B is a synthetic latex of the nitrile or oil-resistant type—especially developed for use in the beater-impregnation of paper.

CHEMIGUM LATEX 245B is easily incorporated into paper pulp during normal papermaking procedures. There's no need for complicated preparations or revisions of methods—just add the latex as received to the beater after the pulp has been beaten. Coagulation of the rubber particles onto the paper fibers is accomplished by the straightforward addition of alum to the beater. Moreover, the incorporation of CHEMIGUM LATEX 245B does not substantially alter the sheet-making properties of the pulp and the usual methods can be used.

The beater addition of CHEMIGUM LATEX 245B has the advantages of conforming to normal papermaking methods and making possible the production of both light and heavy sheets uniformly impregnated with rubber.

The properties imparted to the paper sheets by impregnation with CHEMIGUM LATEX 245B include greatly improved flexibility, greater elongation and higher edge-tear strength. Latex impregnated papers are particularly adaptable to the manufacture of artificial leather products such as book covers, brief cases, folders, luggage and other specialty items.

For full details and samples for evaluation, write today to:
Goodyear, Chemical Division, Akron 16, Ohio.

GOODYEAR

We think you'll like "THE GREATEST STORY EVER TOLD"—Every Sunday—ABC Network

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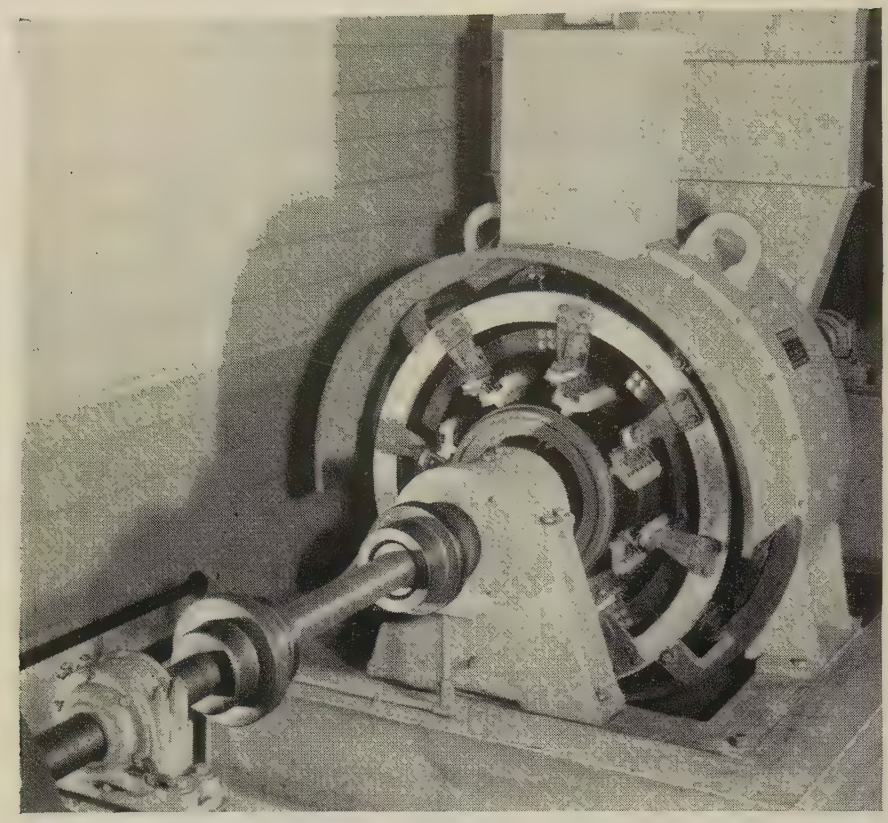
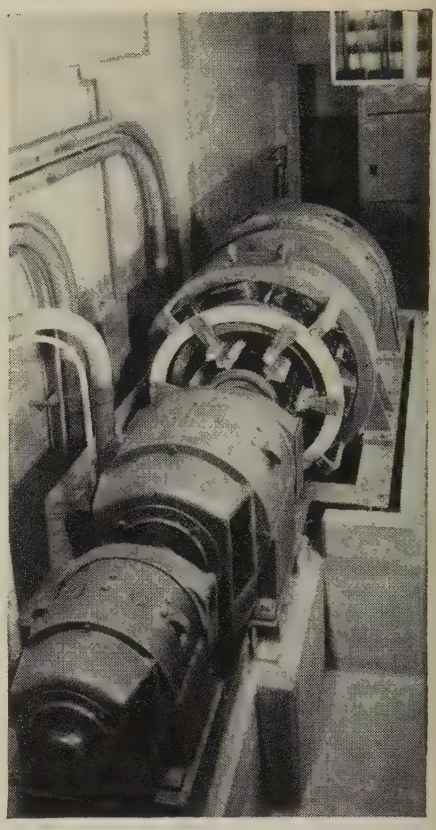
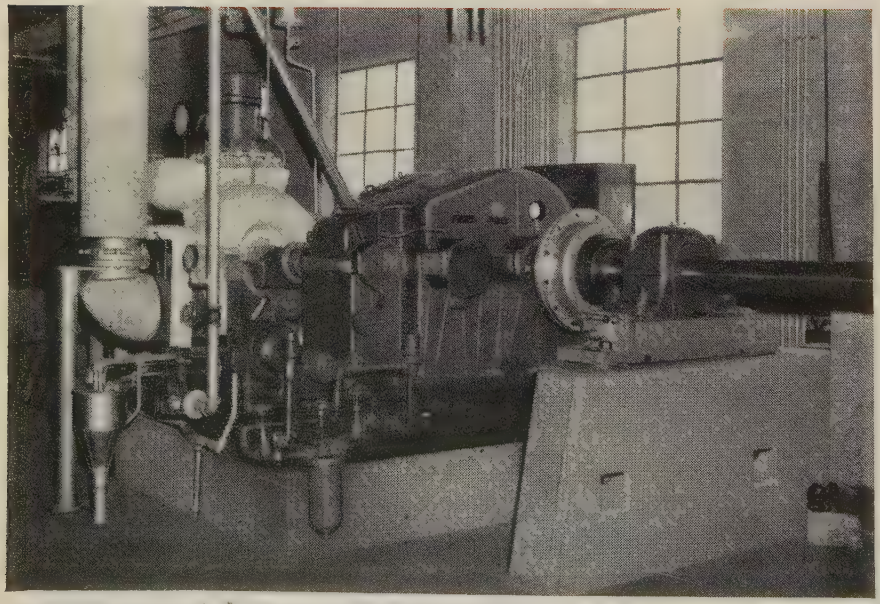


Regardless of load changes, any pre-set speed can be kept with pinpoint accuracy by G-E single-motor drives for lineshaft-driven machines. Simple, flexible operation—with speed ranges up to 10:1 or higher—helps maintain uniform weight and quality of the sheet. At the Camas, Wash., paper mill of the Crown-Zellerbach Corp., this G-E main drive motor for the No. 15 paper machine is rated 900 hp, 600 rpm, 600 volts.



Supplying power to motor at left is this G-E main motor-generator set, including 750-kw d-c generator, 1250-hp synchronous induction motor, 150-kw auxiliary generator, and 25-kw d-c exciter. All are mounted on a common base for easy installation.

Get more paper from your



Reliably, safely, at low cost—G-E mechanical drive-turbines can provide your lineshaft machine with mechanical power as a by-product of your process steam. Adjustable speed with ranges up to 10:1 or higher are provided with great accuracy by their governors. At the Lewiston, Idaho, paper mill of Potlatch Forests, Inc., one of these G-E units—rated 1750 hp, 7000/450 rpm—is shown driving a paper-machine lineshaft.

CO-ORDINATED PAPER-MILL DRIVES

NEW! G-E Motor Selection and Application Course—timed to meet today's needs—can help train your paper-mill employees, increase their efficiency. A G-E "More Power to America" program, it shows how motors work, types now in use, how to select and apply them for specific jobs. Bulletin GEA-4938-16 describes the course in full. ***Write for it!***

For smaller horsepower drives, easy low-cost installation is one big reason why more and more paper-mill operators are using the G-E Speed Variator single-motor drive. An adjustable-voltage d-c drive operating from a-c power, it includes a packaged power unit that's easy to install, maintain, and re-locate if necessary. At the Bay State paper mill of Crane & Company, Inc., Dalton, Mass., the compact, enclosed Speed Variator power unit—shown here behind its G-E 40-hp d-c motor on a lineshaft-driven paper-making machine—"makes for smoother operation all the way through," say company engineers.

lineshaft-driven machines!

One of these General Electric drives—correctly engineered to your needs—can bring out the best in your lineshaft-driven machines, increase your output of uniform-quality paper!

Which drive will do the most for *your* lineshaft-driven paper machines—in raising output to your paper machine's top capacity, maintaining a uniform high-quality sheet, and keeping your paper-making costs down?

It's a problem that can't be solved the same way every time. That's why General Electric offers paper-mill operators a choice of lineshaft drives shown on these pages. With this variety—and with an experienced G-E paper-industry engineering specialist helping in your selection and application problems every step of the way—you're sure of an answer that's *right for you.*

It may call for holding desired speed with great accuracy, either with a G-E single-motor drive or with an efficient G-E mechanical drive turbine to generate mechanical power and supply process steam. In either case, you'll get the right combination of speed-control and sensitivity, the speed range desired, and a drive that's easier to operate, provides top reliability, and requires less maintenance.

To get more paper from your lineshaft-driven machines, get in touch with your G-E paper-mill specialist—now!—at your nearest G-E office. Meanwhile, send for Bulletin GEA-5288 on single-motor drives. *General Electric Company, Schenectady 5, N. Y.*

GENERAL ELECTRIC

655-10

TAPPI Annual Meeting Program

Commodore Hotel, New York, N. Y., February 18–21, 1952

MONDAY, FEBRUARY 18, 1952

10:00 a.m. First General Session (East Ball Room)

K. P. Geohegan, President of TAPPI, Howard Paper Mills, Inc., Dayton, Ohio, *Chairman*

1. Secretary's Report, by R. G. Macdonald.
2. President's Address, by K. P. Geohegan.
3. Report of Tellers Committee.
4. Report of Joint Text Book Committee, by J. N. Stephenson.
5. "The Overall Water Supply Situation Throughout the United States Pulp and Paper Regions," by Fred H. Klaer, Jr., Ranney Method Water Supplies, Inc., Columbus, Ohio.

9:15 a.m. TAPPI-ASTM Technical Committee on Petroleum Wax (1) Room F

9:15 a.m. TAPPI-ASTM Technical Committee on Petroleum Wax (8) Room G

2:00 p.m. Committee Meetings (Grand Ball Room)

All committee meetings will be held in the Grand Ball Room unless otherwise indicated. Any member of the Association may attend any committee meeting. If you are not at present a member of a committee please introduce yourself to the Chairman of the committee that you wish to attend. Tables assigned to committees will be indicated by poster cards.

Research Development Division (Grand Ball Room)

W. F. Holzer, Crown Zellerbach Corp., Camas, Wash., *General Chairman*

Statistics Committee—J. F. Langmaid, Jr., *Chairman*
Microbiological Committee—R. F. DeLong, *Chairman*

Industrial Division (Grand Ball Room)

R. W. Kumler, American Cyanamid Co., New York, N. Y., *General Chairman*

Nonfibrous Raw Materials Committee—E. N. Poor, *Chairman*
Water Committee—A. S. Erspamer, *Chairman*
Fibrous Agricultural Residues Committee—S. I. Aronovsky, *Chairman*
Structural Fibrous Materials Committee—E. M. Jenkins, *Chairman*
- Test Board Formation Subcommittee—R. C. Lacey, *Chairman*
- Drainage Time Testing Subcommittee—J. J. Perot, *Chairman*
- Transverse and Deflection Testing Subcommittee—J. F. Campbell, *Chairman*
- Water Absorption, Water Penetration, and W. V. P. Subcommittee—A. T. Walton, *Chairman*
- Nail Holding and Racking Test Subcommittee—T. C. Duvall, *Chairman*
- Light Reflection Measurement Subcommittee—W. L. Scott, *Chairman*

Converting and Consuming Division (East Ball Room)

M. L. Downs, Thilmany Pulp & Paper Co., Kaukauna, Wis., *General Chairman*

Graphic Arts Committee—F. D. Elliott, *Chairman*
Coating Committee—J. H. Heuer, *Chairman*
Wet Strength Committee—K. W. Britt, *Chairman*
Plastics Committee—E. C. Jahn, *Chairman* (3:30 p.m.)
- Fabrication and Structural Products Subcommittee—C. J. Straka, *Chairman*
- Plastics in Paper Converting Subcommittee—R. T. Nazarro, *Chairman*
- Resin Analysis Subcommittee—S. E. Church, *Chairman*
- Cellulosic Materials Subcommittee—F. A. Simmonds, *Chairman*

Pulp Manufacture Division (see below)

K. O. Elderkin, Crossett Paper Mills, Crossett, Ark., *General Chairman*

Alkaline Pulping Committee—Henry Vranian, *Chairman* (Room B)
Mechanical Pulping Committee—F. W. O'Neil, *Chairman* (Room B)
Chemical Products Committee—Arthur Pollak, *Chairman* (Tues., 9:15 a.m., Room D)
Paper Deinking Committee—J. J. Forsythe, *Chairman* (Room C)
Pulp Purification Committee—W. D. Harrison, *Chairman* (Room G)
Acid Pulping Committee—J. M. McEwen, *Chairman* (Room B)

Paper Manufacture Division (Grand Ball Room)

R. P. Price, Hammermill Paper Co., *General Chairman*

Preparation of Papermaking Materials Committee—F. S. Klein, *Chairman*
Paper Making (Cylinder) Committee—J. J. Harrison, *Chairman* (Room F)
Paper Making (Fourdrinier) Committee—N. R. Phillips, *Chairman* (Room E)

Testing Division (West Ball Room)

J. d'A. Clark, Consultant, Longview, Wash., *General Chairman*

Nonfibrous Materials Testing Committee—J. P. Casey, *Chairman*
Pulp Testing Committee—L. V. Forman, *Chairman*
Optical Properties Committee—H. E. Obermanns, *Chairman*
Microscopy—C. E. Brandon, *Chairman*
Paper Testing—A. K. Warner, *Chairman*
Packaging Materials Testing—L. E. Simerl, *Chairman*
Container Testing—C. H. Krebs, *Chairman* (Wed. 2:00 p.m., Room F)
Chemical Methods—A. S. O'Brien, *Chairman*
Fibrous Materials Testing—E. R. Schafer, *Chairman*
TAPPI-ASTM Joint Committee on Petroleum Wax (Advisory) A. M. Heald, *Chairman*

Note: Reports of committee chairmen will be given at this session.

Engineering Division (Room A) (2:00 p.m.)

C. J. Sibler, West Virginia Pulp & Paper Co., *General Chairman*, and W. C. Bloomquist, General Electric Co., Schenectady, N. Y., *General Secretary*

Steam and Power Committee—H. R. Arnold, *Chairman*
Materials Handling Committee—A. P. Schnyder, *Chairman*
Chemical Engineering Committee—J. R. Lientz, *Chairman*
Mill Maintenance and Materials Committee—H. F. Parker, *Chairman*
Mill Design and Economic Aspects Committee—R. A. Packard, *Chairman*
Engineering Research and Machine Design Committee—J. D. Lyall, *Chairman*
Electrical Engineering Committee—Ray Foster, *Chairman*
Data Sheets Committee—H. J. Perry, *Chairman*
Drying and Ventilating Committee—M. L. Barker, *Chairman*

TUESDAY, FEBRUARY 19, 1952

9:15 a.m. Structural Fibrous Materials (Room A)

E. M. Jenkins, Johns-Manville Corp., Manville, N. J., *Chairman*

1. "The Use of Asphalt Emulsions in the Manufacture of Insulating Board Products," by J. J. Perot, Flintkote Co., Whippany, N. J.
2. "A Preliminary Investigation of the Adaptability of the General Electric Puncture Tester to the Testing of Struc-

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tural Insulating Board," by J. A. Van den Akker and Keith W. Hardacker, Institute of Paper Chemistry, Appleton, Wis.
3. "Drying of Insulating Boards," by H. C. Jessen, Johns-Manville Corp., Manville, N. J.
4. Report of the Activities of the Structural Fibrous Materials Committee, by E. M. Jenkins, *Chairman*.

9:15 a.m. Water (East Ball Room)

A. S. Erspamer, P. H. Glatfelter Co., Spring Grove, Pa., *Chairman*

1. "The Activation of Silica and Its Use in Treatment of Mill Water Supply, White Water Utilization and Waste Disposal," A. E. Griffin, Wallace & Tiernan, Inc., Belleville, N. J.
2. "Use of Ozone in Water Purification," by Victor Hahn, Welsbach Corp., Philadelphia, Pa.
3. "Filtration of Board Mill White Waters," by J. Howard Wright, S. Austin Bicking Co., Downingtown, Pa.
4. "Discussion of Questionnaire on Analytical Methods and Solids Losses Evaluation Procedures for Pulp and Paper Mill White Water and Effluents," by Arthur M. Thurm, Champion Paper & Fibre Corp., Hamilton, Ohio.

9:15 a.m. Plastics (West Ball Room)

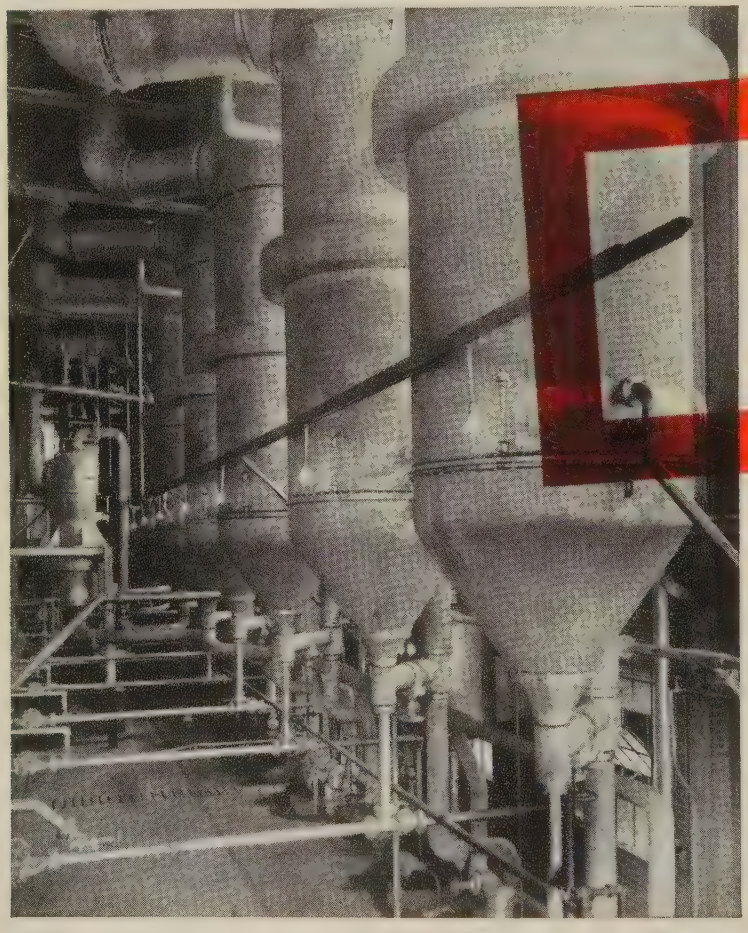
E. C. Jahn, College of Forestry, State University of New York, Syracuse, N. Y., *Chairman*

1. "Acetylation of Cellulose," by R. C. Blume, E. I. du Pont de Nemours & Co., Inc., Waynesboro, Va.
2. "Purified Hardwood Pulps for Chemical Conversion. I. Aspen Sulphite and Prehydrolysis—Sulphate Pulps," by F. A. Simmons, R. M. Kingsbury, J. S. Martin, and E. L. Keller, Forest Products Laboratory, Madison, Wis.
3. "Mechanics of Adding Latex to Paper at the Head Box," by H. G. Bimmerman, E. I. du Pont de Nemours & Co., Inc., Wilmington, Del.
4. "Polyamide Resin Suspensoids—A New Tool for the Paper Converter," by Harold Wittcoff, M. M. Renfrew, and Fred B. Speyer, General Mills, Inc., Minneapolis, Minn.
5. "New Methods of Adding Neoprene Latex to Pulp," by R. H. Walsh, E. I. du Pont de Nemours & Co., Inc., Wilmington, Del.

9:15 a.m. Statistics (Room B-C)

John F. Langmaid, Jr., S. D. Warren Co. Cumberland Mills, Me., *Chairman*

1. "A Printer's Interest in Statistical Quality Control in



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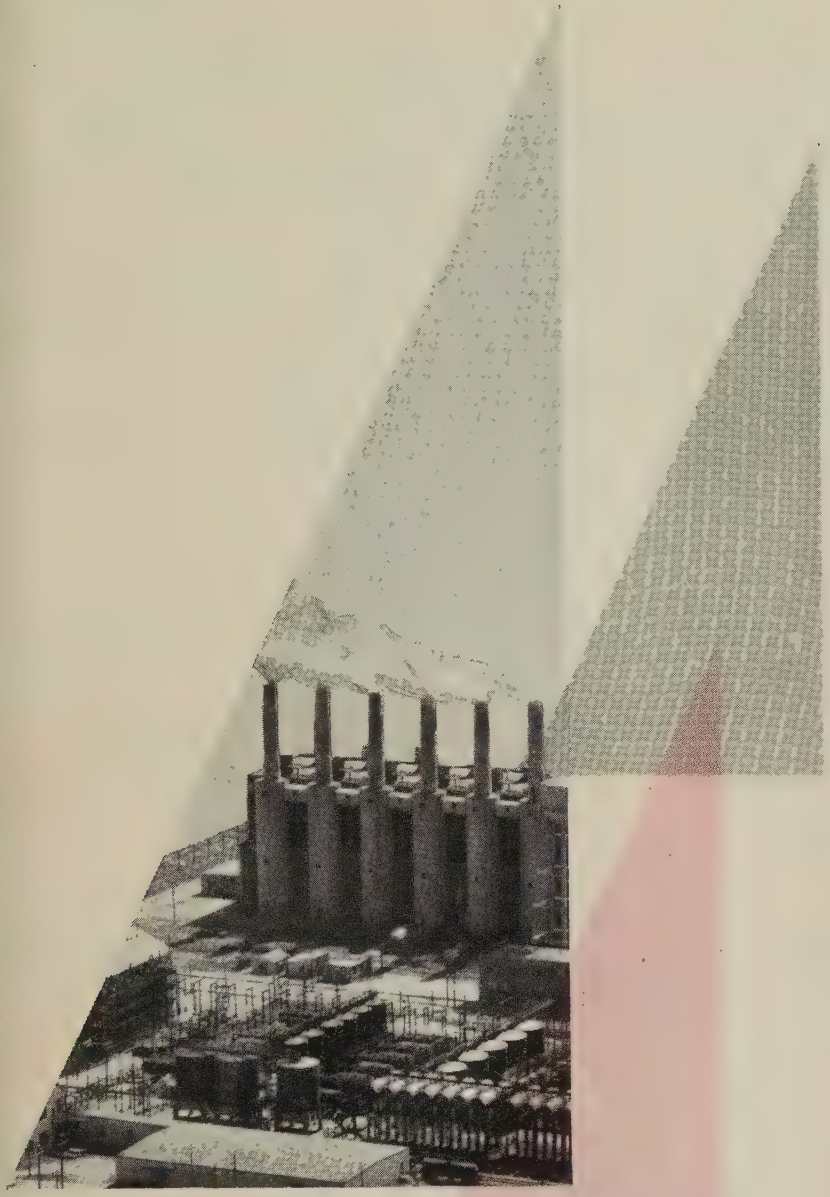

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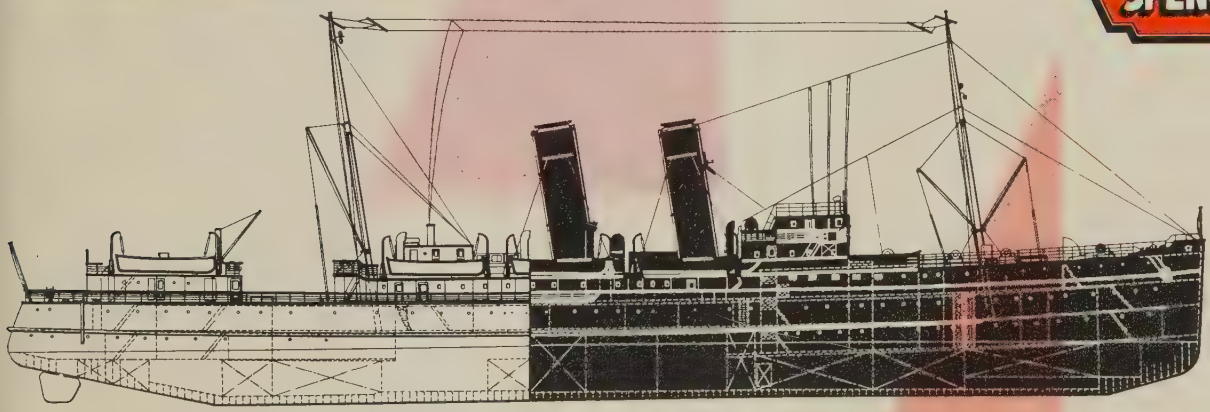

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Paper Mills," by O. H. Somers, Western Printing & Lithographing Co., Racine, Wis.
2. "Significance of Laboratory Sizing Data," by Ell Dee Compton, Monsanto Chemical Co., Everett, Mass.
3. "Management Reviews a Controlled Operation," by W. A. MacCrehan, Empire Box Co., Garfield, N. Y.
4. "Case Studies in the Application of Industrial Statistics, Part II," by Charles A. Bicking, U. S. Dept. of Defense, Washington, D. C., and R. T. Trelfa, Watervliet Paper Co., Watervliet, Mich.

9:15 a.m. Corrugated Containers (Grand Ball Room)

F. D. Long, Container Corp. of America, Chicago Ill., *Chairman*

Symposium on "Corrugated Starch Adhesives," Burt Mendlin, Cornell Paperboard Co., Milwaukee, Wis., *Moderator.*

1. "Résumé of Replies to Starch Adhesives Questionnaire," by Burt Mendlin.
2. "Starch Formulations and Control of Starch Viscosities," by J. E. Killinger, Penick & Ford, Inc., Cedar Rapids, Iowa.
3. "Advantages and Disadvantages of One vs. Two Starch Systems," by E. S. Gantt, Corn Products Sales Co., Chicago, Ill.
4. "Fluff, Flutter, Fingerlines and Other Operational Problems," by S. Drum, Container Corp. of America, Chicago, Ill.
5. "Heat Balance on a Corrugator," by R. T. Cassady, Jackson Box Co., Cincinnati, Ohio.
6. "Testing and Quality Control," by D. P. McNelly, Fort Wayne Corrugated Paper Co., Fort Wayne, Ind.
7. Panel Discussion of Papers 1–6 (speakers will constitute panel).

9:15 a.m. Papermaking (Fourdrinier) (Room G)

N. R. Phillips, Champion Paper & Fibre Co., Canton, N. C., *Chairman*

1. Round-Table Discussion on the "Paper Machine Shake."

9:15 a.m. Chemical Products (Room D)

Arthur Pollak, Consulting Engineer, New York, N. Y., *Chairman*

1. Committee Meeting.

9:15 a.m. TAPPI-ASTM Technical Committee on Petroleum Wax (2) (Room E)

9:15 a.m. TAPPI-ASTM Technical Committee on Petroleum Wax (3) (Room F)

2:00 p.m. Preparation of Papermaking Materials (East Ball Room)

F. S. Klein, Byron Weston Co., Dalton, Mass., *Chairman*

1. "Mechanical Correction of Two-Sidedness," by M. J. Landberg and P. R. Schreiber, General Dyestuff Corp., and H. H. Behrens, Irvin Dyestuff Corp., Ltd. (Canada).
2. "The Use of Graphic Panels in the Paper Industry," by Donald B. Scrivens, Taylor Instrument Co., Rochester, N. Y.
3. "The Beaterpulper," by Arthur W. Infanger, Cowles Co., Cayuga, N. Y.
4. "Handling Materials in the Paper Industry," by L. O. Millard, Link-Belt Co., Chicago, Ill.
5. "The Hammermill Dirt Separator," by E. L. Rastatter and A. H. Croup, Hammermill Paper Co., Erie, Pa.

2:00 p.m. Acid Pulping (Room B-C)

John M. McEwen, Weyerhaeuser Timber Co., Pulp Div., Everett, Wash., *Chairman*

1. "Pilot Plant Recovery of Heat and Sulphur from Spent Ammonia Base Sulphite Pulping Liquor," by J. H. Hull, Crown Zellerbach Corp., Camas, Wash., and G. V. Palmrose, Soundview Div. Scott Paper Co., Everett, Wash.
2. "Acid Pulping Committee Report on TAPPI Strainer and Circulation Survey," by John M. McEwen, Pulp Div., Weyerhaeuser Timber Co., Everett, Wash.
3. "Sulphite Digester Lining Problems," by William H. Kraske, Oxford Paper Co., Rumford, Me.
4. "Magnesia-Base Recovery for the Sulphite Pulping Industry," Svarre Hazelquist, Pulp Div., Weyerhaeuser Timber Co., Longview, Wash., and Charles E. Rogers, Babcock & Wilcox Co., Alliance, Ohio.
5. "The Manufacture and Properties of Chlorine Dioxide," by E. Kesting, Elektrochemische Werke, Munich, Germany.
6. "Soda-Base Sulphite Recovery by the Sulfox Process," by Arthur Pollak, Consulting Engineer, New York, N. Y., and Robert S. Aries, Polytechnic Institute of Brooklyn, Brooklyn, N. Y.
7. "Modern Developments in Welded Barking Drum Design," by George M. Dick, Canadian Ingersoll-Rand Co., Sherbrooke, Que.

2:00 p.m. Fundamental Research (West Ball Room)

Harry F. Lewis, Institute of Paper Chemistry, Appleton, Wis., *Chairman*

1. "Physical Nature of Hemicelluloses" (Institute of Paper Chemistry).
2. "Effect of Various Constituents of Wood and Pulp on the Ignition Point of the Material" (Institute of Paper Chemistry).
3. "A Theory of Alkaline Peroxide Bleaching," by D. M. Martin, Cornell Paperboard Products Co., Milwaukee, Wis.

2:00 p.m. Corrugated Containers (Grand Ball Room)

F. D. Long, Container Corp. of America, Chicago, Ill., *Chairman*

Symposium on "Corrugated Silicate Adhesives," M. F. Knack, River Raisin Paper Co., Monroe, Mich., *Moderator.*

1. "Résumé of Replies to Silicate Adhesives Questionnaire and Remarks on Efficient Use of Silicate," by E. G. Boyce, E. I. du Pont de Nemours & Co., Inc., Wilmington, Del.
2. "Effect of Varying Compositions of Silicate on Corrugator Operation," by R. L. Kreyling, Philadelphia Quartz Co., Philadelphia, Pa.
3. "Proper Mechanical Adjustment for Good Corrugator Operation," by C. Kreegor, Industrial Corrugated Box Co.
4. "Clearances on the Corrugator," by Charles Mitchie, Samuel Langston Co., Camden, N. J.
5. "Steam Supply and Evacuation," by J. E. Wilske, Owens-Illinois Glass Co., Alton, Ill.
6. Panel Discussion of Papers 1–5 (speakers will constitute panel).

2:00 p.m. Microbiological (Room A)

R. F. De Long, Marathon Corp., Menasha, Wis., *Chairman*

1. "A Method of Laboratory Evaluation of Chemicals as Potential Slimicides," by S. I. Cohen and Marcine Kahn, Gallowher Chemical Corp., New York, N. Y.
2. "Performance of Paper Machine Wet Felts. II. Further Studies of the Plugging of Felts," by D. W. French, C. M. Christensen, and S. J. Buckman, Buckman Laboratories, Memphis, Tenn.
3. "Lever Spore Cloud Method for the Evaluation of Antimycotic Properties of Paper and Paperboard," by L. S. Vinson, Lever Bros. Co., Cambridge, Mass.

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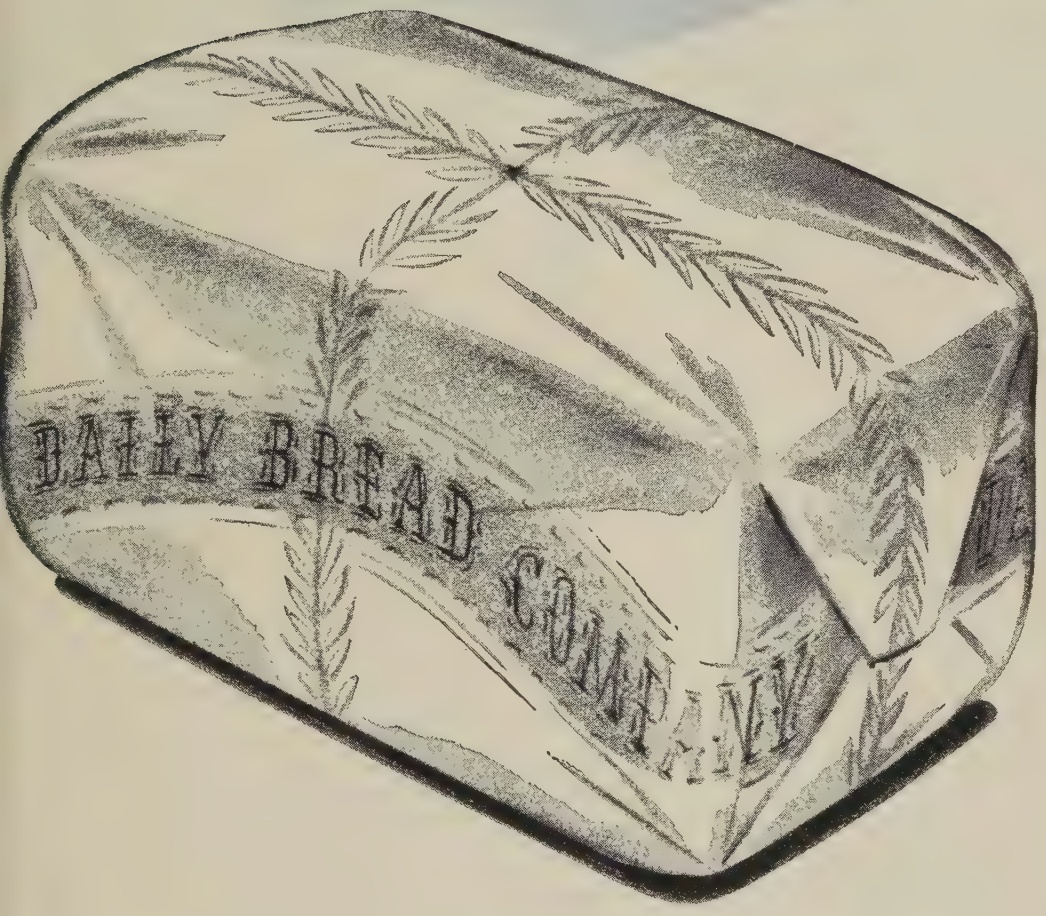

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4. "Factors Affecting Slime Growth," by L. A. McKeown, Consultant, Plattsburg, N. Y.
5. "The Effect of Various Pulps on the Biological Activity of Phenyl Mercuric Acetate," by B. F. Shema, Institute of Paper Chemistry, Appleton, Wis.

2:00 p.m. Hydraulics (Room D)

K. J. Mackenzie, Eastman Kodak Co., Rochester, N. Y., *Chairman*

1. Committee Meeting.

2:00 p.m. TAPPI-ASTM Technical Committee on Petroleum Wax (5) (Room E)

2:00 p.m. TAPPI-ASTM Technical Committee on Petroleum Wax (7) (Room F)

2:00 p.m. Structural Fibrous Materials (Room G)

Edward M. Jenkins, Johns-Manville Corp., Manville, N. J., *Chairman*

1. Committee Meeting.

WEDNESDAY, FEBRUARY 20, 1952

9:15 a.m. Graphic Arts (East Ball Room)

Fred D. Elliott, Time, Inc., Springdale, Conn., *Chairman*

1. "Review of the New Developments in the Graphic Arts," by R. H. Simmons, Government Printing Office, Washington, D. C.
2. "Mechanization of Sheet Paper Counting," by Charles Stevens, Charles Stevens, Inc., Toledo, Ohio.
3. "The Relation Between Paper and Gravure Printing," by R. T. Van Derveer, Triangle Publications, Philadelphia, Pa.
4. "Pick Testing of Offset Papers," by Gordon C. Wheeler and Robert F. Reed, Lithographic Technical Foundation, Chicago, Ill.
5. Panel Discussion on "Measurement of Printing Smoothness": S. M. Chapman, Optical Supervisor, Printability Section, Pulp and Paper Research Institute of Canada, Montreal, P. Q.; Kenneth Latimer, Time, Inc., Springdale, Conn.; G. R. Sears, Institute of Paper Chemistry, Appleton, Wis.

9:15 a.m. Wet Strength (Grand Ball Room)

Kenneth W. Britt, Scott Paper Co., *Chairman*

1. "Methods of Increasing the Efficiency of Wet-Strength Resins," by J. B. Davidson, Plaskon Div., Libbey-Owens-Ford Glass Co., Toledo, Ohio.
2. "Problems in Testing Wet-Strength Paper," by C. S. Maxwell, American Cyanamid Co., Stamford, Conn.
3. "A Mill Control Method for Wet-Strength Papers," by A. G. Durgin and W. G. Baker, Brompton Pulp & Paper Co., East Angus, P. Q.
4. Round-Table Discussion of "Wet-Strength Testing Methods."

9:15 a.m. Alkaline Pulping and Chemical Engineering (West Ball Room)

Henry Vranian, Chesapeake Corp., West Point, Va., and J. R. Lientz, Union Bag & Paper Corp., Savannah, Ga., *Chairmen*

1. "Effect of Chip Size in Kraft Pulping," by W. J. Nolan and Wilson F. Brown, University of Florida, Gainesville, Fla.
2. "Studies in Continuous Alkaline Pulping II, High Yield Cellulose Pulps from Scrub Oak," by W. J. Nolan and W. F. Brown, University of Florida.
3. "The Debarking of Scrub Oak," by R. L. Harvin, W. J. Nolan, and W. F. Brown, University of Florida.
4. "Esterification of Tall Oil with Glycerine," by Robert S. Aries and Melvin Wolkstein, Polytechnic Institute of Brooklyn, Brooklyn, N. Y.
5. "Uniform Cooking by Continuous Kraft Semichemical Process," by A. P. Yundt, Camp Mfg. Co., Inc., Franklin, Va.
6. Paper by E. M. Leavitt, North Carolina Pulp Co., Plymouth, N. C.

9:15 a.m. Papermaking (Cylinder) (Room A)

James J. Harrison, Michigan Carton Co., Battle Creek, Mich., *Chairman*

1. Round-Table Discussion on "Wet End Forming and Pressing on Cylinder Machines."

9:15 a.m. Papermaking (Fourdrinier) (Room E)

Noel R. Phillips, Champion Paper & Fiber Co., Canton, N. C. *Chairman*

1. Round Table Discussion of Paper Defects.

9:15 a.m. TAPPI-ASTM Technical Committee on Petroleum Wax (4) (Room F)

9:15 a.m. TAPPI-ASTM Technical Committee on Petroleum Wax (6) (Room G)

2:00 p.m. Packaging Materials and Containers (East Ball Room)

L. E. Simerl, Marathon Corp., Menasha, Wis., *Chairman*

1. "Arganoleptic Evaluation of Paper Products for Food Packaging," by L. C. Cartwright, Foster D. Snell, Inc., New York, N. Y.
2. "A Volumetric Gas Analysis Method for Measuring Simultaneously Oxygen and Carbon Dioxide Permeabilities," by Arthur H. Landrock and Bernard E. Proctor, Massachusetts Institute of Technology, Cambridge, Mass.
3. "Development of Design Data for Corrugated Fiberboard Shipping Containers," by K. Q. Kellicutt and E. F. Landt, Forest Products Laboratory, Madison, Wis.
4. "Some Observations on the Kinetic Coefficient of Friction of Paper," by Geoffrey Broughton, Lowell Textile Institute, Lowell, Mass., and Joan L. Gregg, U. S. Envelope Co., Worcester, Mass.

2:00 p.m. TAPPI-ASTM Technical Committee on Petroleum Wax (Room B-C)

A. M. Heald, Scott Paper Co., Chester, Pa., *Chairman*

Reports of Subcommittees:

1. Strength Tests, J. W. Padgett, *Chairman*
2. Flow Tests, Hans Schindler, *Chairman*
3. Stability Tests, G. P. Hinds, Jr., *Chairman*
4. Sensory Tests, G. D. Fronmueller, *Chairman*
5. Composition, J. B. Rather, Jr., *Chairman*
6. Blocking, M. W. Kane, *Chairman*
7. Sealing and Laminating, G. C. Borden, Jr., *Chairman*
8. Scuff-Glass, G. P. Mueller, *Chairman*

2:00 p.m. Alkaline Pulping and Chemical Engineering (West Ball Room)

Henry Vranian and J. R. Lientz, *Chairmen*

Symposium on "Semichemical Pulping."

1. "Semichemical Process Flow Sheets," by J. E. Sapp, Gaylord Container Co., Bogalusa, La.
2. "Corrosion Problems in Semichemical Pulping," by H. O. Teeple, International Nickel Co., New York, N. Y.
3. "Semichemical Liquor Preparation and Recovery," by R. P. Whitney, Institute of Paper Chemistry, Appleton, Wis.
4. "Semichemical Pulping of New England Woods," by Daniel J. Adams, Bird Machine Co., East Walpole, Mass.
5. "Semichemical Pulping—A Process Study," by C. W. Converse, Sprout, Waldron & Co., Muncy, Pa.

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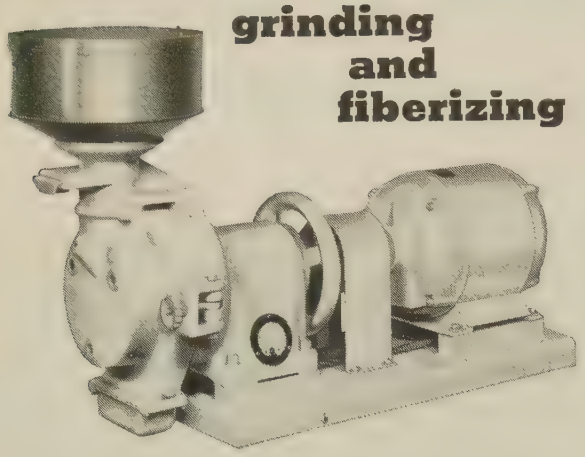
Bauer 8" Lab. Mill for grinding and fiberizing



This mill is equipped with a 3-hp to 10-hp built-in motor or a V-sheave. It has a revolving disc and stationary disc. Built with the precision required for laboratory use, the mill has ample stamina for production service.

The model illustrated is especially adapted for reducing and fiberizing wet materials such as samples of semi-cooked chips. The front case is readily opened for recovery of the complete sample and for cleaning.

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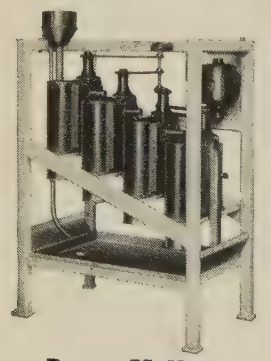
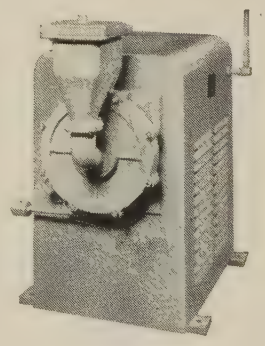


Bauer-McNett Classifier

Standard apparatus for fractionating samples of pulp or fluid paper stock to classify fiber lengths, providing basic data for the regulation of beating or refining operations.



Mead Laboratory Beater

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ASK FOR LITERATURE

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2:00 a.m. Papermaking (Fourdrinier) (Grand Ball Room)

NOEL R. PHILLIPS, Champion Paper & Fibre Co., Canton, N. C., *Chairman*

1. "Straw Pulp-Wood Pulp Blends for Various Types of Papers," by S. I. Aronovsky and A. J. Ernst, Northern Regional Research Laboratory, Peoria, Ill., and R. J. Seidl and R. M. Kingsbury, Forest Products Laboratory, Madison, Wis.
2. "Fundamentals of Lithography for Paper Makers," by R. F. Reed, Lithographic Technical Foundation, Chicago, Ill.
3. "Curl," by A. S. Erspamer and W. D. Rice, P. H. Glatfelter Co., Spring Grove, Pa.

2:00 p.m. Papermaking (Fourdrinier) (Room G)

L. P. THOMPSON, Personal Products Corp., Milltown, N. J., *Chairman*

1. Round-Table Discussion on "Tissue Paper Manufacture."

2:00 p.m. ASTM-TAPPI Joint Committee on Container Testing (Room F)

E. R. STIVERS, Stapling Machines Co., Rockaway, N. J. *Chairman*

1. Committee Meeting.

THURSDAY, FEBRUARY 21, 1952

9:00 a.m. Chemical Methods and Pulp Testing (West Ball Room)

A. S. O'BRIEN, Eastman Kodak Co., Rochester, N. Y., and L. V. FORMAN, Scott Paper Co., Chester, Pa., *Chairman*

1. "The Determination of Copper in Wood Pulp with Tetraethylenepentamine," by R. M. Kingsbury and C. L. Lake, Forest Products Laboratory, Madison, Wis.
2. "Determination of Pentosans in Highly Purified Wood Pulp," by Edgar D. Smith, and L. N. Rogers, Buckeye Cotton Oil Co., Memphis, Tenn.
3. "A Colorimetric Method for the Determination of Pentosan Content of Highly Refined Pulps," by T. Davis and A. S. O'Brien, Eastman Kodak Co., Rochester, N. Y.
4. "On Sampling of Wood Pulp for Moisture," by A. P. Bollinghausen, Kimberly-Clark Corp., Neenah, Wis.
5. "Routine Determination of Lignin by Modified Tingle Procedure," by B. L. Browning and L. O. Bublitz, Institute of Paper Chemistry, Appleton, Wis., and Paul E. Trout, P. A. Franckowiak, and J. L. Hamm, American Box Board Co., Filer City, Mich.

9:00 a.m. Papermaking (Cylinder) (Room A)

JAMES J. HARRISON, *Chairman*

1. Round-Table Discussion on "Stock Cleaning and Refining of Waste Paper for Use on Cylinder Machines."

9:00 a.m. Fibrous Agricultural Residues (Room B-C)

S. I. ARONOVSKY, Northern Regional Research Laboratory, Peoria, Ill., *Chairman*

1. Round-Table Meeting on "Straw Utilization Problems."

1:00 p.m. Annual Luncheon (Grand Ball Room)

KENNETH P. GEOHEGAN, President TAPPI, *Presiding*

1. Introduction of Guests.
2. Presentation of the TAPPI Medal to Raymond S. Hatch, Hudson Pulp & Paper Co., Palatka, Fla., by Robert B. Wolf, Consultant, New Canaan, Conn.
3. "Scientific and Technical Manpower Problems," by Walter J. Murphy, Editor, *Industrial and Engineering Chemistry*, Washington, D. C.

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Magnesium-Base Sulphite Pulping

WILLIAM Q. HULL, R. E. BAKER, and C. E. ROGERS

IN THE world's first sulphite pulp mill, constructed by the Swedish chemist, C. D. Ekman, at Bergvik, Sweden, in 1874, magnesium bisulphite was used for a period of time as a base for the preparation of the cooking acid (*1*). As the process developed and came into general use, however, limestone was almost universally adopted because of its widespread availability and low cost. In relatively recent years magnesia-base pulping has again received serious consideration and the first full-scale commercial plant utilizing the process is in operation.

The sulphite process is second only to kraft as a source of chemical wood pulp; production by the sulphite process in the United States in 1950 was approximately 3,000,000 tons (Table I). Its origin is generally attributed to the efforts of Tilghman, who was granted a patent in 1876 entitled, "Treating Vegetable Substances for Making Paper Pulp" (*10*). Tilghman originally proposed lead-lined, cylindrical vessels as digesters and numerous mechanical difficulties resulted. Ekman, presumably unaware of the Tilghman patents, equipped his mill with small rotating digesters heated indirectly by means of steam jackets. During this same period, A. Mitscherlich developed a process in Germany using horizontal, stationary cylindrical digesters, indirectly heated by coils of lead pipe, in the place of Ekman's rotating vessels, and Eugene Ritter and Carl Kellner, in Austria, operated mills in which steam was admitted directly into the cooking vessel. The first sulphite mill in the United States, built by Charles S. Wheelwright in East Providence, R. I., was based on Ekman's work. A few years later, in 1887, the first Mitscherlich mill in this country was constructed in Alpena, Mich.

Today, chemical pulp is made by three basic processes—kraft, sulphite, and soda. In earlier reports in this series, the history and manufacture of kraft pulp and paper were described in detail, and a comparison of process and product characteristics of the three basic methods of chemical pulpmaking was given (*7, 8*). This article describes the manufacture of pulp by the sulphite process and more especially sulphite pulping in which magnesium oxide is used as the base of the cooking liquor.

ECONOMICS OF SULPHITE PULPING

All chemical pulp making involves the separation of cellulose, which serves as raw material, from the unstable and unwanted lignin and other noncellulosic components of the wood. In the kraft process, a mixture of sodium hydroxide and sodium sulphide is used to dissolve noncellulosic materials from the wood. The soda process depends on the dissolving action of sodium hydroxide alone. The sulphite process differs from these alkaline processes in attaining the same action with an acid cooking liquor.

The more common sulphite process consists of the digestion of wood, which has been reduced to chips, in an aqueous solution containing calcium bisulphite, $Ca(HSO_3)_2$, and an excess of sulphur dioxide at temperatures from 130° to 150°C. In order that the chips be completely impregnated with cooking liquor, the temperature is raised slowly during the first few hours of the cook. A cooking time of from 7 to 12 hours is required. The pulping process involves two principal types of reactions, which are probably concurrent: the reaction of the lignin with the bisulphite and the hydrolytic splitting of the cellulose-lignin (*9*). Hemicelluloses are hydrolyzed to simpler soluble compounds, and the extraneous wood components are also acted on. Not dissolved is the resistant cellulose which actually is sulphite wood pulp. This is separated from the cooking liquor and processed into a finished pulp product. The cooking liquor is prepared from sulphur dioxide gas (obtained from burning sulphur), which is absorbed in water in the presence of limestone to form calcium bisulphite.

The cellulose content of various woods in the United States ranges from 51 to 65% (*4*), and since this is the primary constituent that is desired in the end pulp product, it follows that approximately half of the wood charged is dissolved by the cooking liquor. The composition of the dissolved fraction varies with the specific process used, but, generally, the chief organic components are modifications of lignin and the more readily dissolved carbohydrates and their modifications. The by-product liquor resulting after the separation of the cellulose is called sulphite residual liquor or red liquor. It has been estimated that for every ton of sulphite pulp produced, there result 2000 gallons of the waste liquor containing 12% solids (*5*). Of the solids, as much as 65% is lignosulphonic acid; 20%, reducing sugars; 8.4%, sugar-sulphur dioxide derivatives; and 6.7%, calcium. The composition varies and depends on the wood used and cooking conditions.

In the United States and Canada, about 4,000,000 tons of sulphite pulp are produced annually. Conse-

Table I. U. S. Sulphite Pulp Production (*13*)

Year	Thousand short tons		
	Bleached	Unbleached	Total
1941	1703	1216	2919
1942	1717	1213	2930
1943	1553	883	2436
1944	1523	863	2386
1945	1544	816	2360
1946	1692	784	2476
1947	1902	894	2796
1948	1909	902	2811
1949	1829	707	2536
1950	2111	744	2855

WILLIAM Q. HULL, Associate Editor, *Industrial and Engineering Chemistry*, in collaboration with R. E. BAKER, Weyerhaeuser Timber Co., Longview, Wash., and C. E. ROGERS, The Babcock & Wilcox Co., Alliance, Ohio.

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quently, the volume of waste liquor is enormous, and the dissolved components of the wood weigh approximately 4,900,000 tons. The disposal of this vast amount of organic matter has become a problem in nearly every area where sulphite pulp is produced, particularly because some of the sugars and organic acids become consumers of oxygen and thus pollute streams into which the waste liquor is directed.

During the past 15 years interest in the disposition of sulphite waste liquor has been stimulated considerably by public reaction and industrial and legal efforts to eliminate stream pollution of all types. Since more than half of the raw material entering the pulping process appears in the waste liquor as dissolved organic solids, the economic aspect has also encouraged extensive study. Investigations of the waste liquor problem have thus followed two lines of endeavor—disposal and utilization.

In order that there be relatively little decrease in the dissolved oxygen content in waters into which sulphite waste from mills is discharged, waste liquor disposal has had as its objective the reduction of the biological oxygen demand of the waste liquor. Included in these studies have been the use of trickling filters, reaeration, or chemical alteration of the carbohydrate portion of the liquor. All these methods require equipment, power, and maintenance, and result in a higher cost of the pulp produced.

None of the suggested treatments seeks to recover the carbohydrate portion of the waste liquor. Utilization of the solids in the waste liquor has been approached by many who have endeavored to convert the organic material into useful chemical compounds or to extract from the liquor, by precipitation or other means, the lignin or carbohydrates in such a manner that they will find commercial outlets. Varying degrees of success have resulted. Among the principal products which have been investigated or proposed are alcohol by fermentation of the waste liquor; yeast produced by the fermentation process; vanillin from the lignin present; oxalic acid; tanning material; road binders; special cements; Portland cement accelerator; core-binders; and plastics from the lignin present. A recent report in this series described the production of food yeast from sulphite liquor (*3*).

Another important consideration is the heat value of tne dry organic solids in the waste liquor which ranges between 7000 and 8000 B.t.u. per pound. This material, under proper conditions, offers a valuable source of energy, in the form of fuel, which might be supplied to convert wood into chemical pulp. Some years ago, it was realized that if, in the process of utilizing the organic solids as a source of energy, it was possible to recover the chemicals used for producing the pulp, there would result a cyclic process in which all of the resultant products could be utilized by the pulp manufacturer. Attempts to develop such a process brought forth numerous proposals to evaporate the sulphite liquor from calcium-base pulping and use the concentrated liquor as fuel. Approximately 9000 to 10,000 pounds of steam can be produced from the waste liquor resulting from the production of 1 ton of sulphite pulp. However, these proposals were not particularly attractive except as a means of waste liquor disposal because the digesting chemicals are not recovered in a form to permit their re-use in the process. Calcium and sulphur are recovered as calcium sulphate, and as the calcium sulphate cannot be broken down into calcium oxide during the use of the concentrated liquor as a fuel, its recovery as a cooking chemical is not possible.

Sodium and ammonia have been substituted for calcium as a pulping base and are used in a limited number of commercial plants. However, the recovery of the bases as usable chemicals for the sulphite pulping process is complicated and costly.

Trade Glossary

Total sulphur dioxide: Total SO_2, determined by titration according to TAPPI Standard T 604 m, expressed as per cent total SO_2

Free sulphur dioxide ("available" SO_2): Sum of actual free SO_2 plus one half the SO_2 combined as bisulphite, determined by titration according to TAPPI Standard T 604 m, expressed as per cent free SO_2

Combined sulphur dioxide ("nonavailable" SO_2): SO_2 combined with base as monosulphite, determined by difference of free SO_2 and total SO_2, expressed as per cent combined SO_2

Sulphur dioxide: SO_2 gas made by burning sulphur or pyrites with sufficient air to form SO_2

Bisulphite liquor base: Magnesium oxide used in preparation of bisulphite cooking liquors

System of finished acid: Bisulphite liquor prepared by combination of SO_2 and base in presence of water, with excess of SO_2 in solution

Storage acid (also known as raw acid): System acid in reserve for transfer to acid accumulators for fortification by injection of additional SO_2

Accumulator acid: Storage acid held in pressure vessels during fortification by SO_2 in digester-relief gas

Cooking liquor: Accumulator acid ready for use in the digester cooking operation, after fortification is completed

Digester liquor: Cooking liquor as it exists in digester at various stages during progress of cooking operation

Digester to relief: Gas withdrawn from top of digester during early stage of cooking operation and returned to accumulator to conserve SO_2 and heat

Digester side relief: Digester liquor withdrawn by side relief from upper section of digester in early stages of digestion and returned to accumulator or storage to conserve SO_2 and heat

Digester on-side test: Test of digester liquor for total, free, and combined SO_2 just before digester is put on side relief

Digester off-side test: Test of digester liquor for total, free, and combined SO_2 just after digester is taken off side relief

Digester blow-down test: Test of digester liquor for total, free, and combined SO_2 at beginning of pressure-relief period toward end of digestion

Digester dump test: Test of digester liquor for total, free, and combined SO_2 just before digester is dumped

Sulphite waste liquor or red liquor: Spent liquor from sulphite digestion (containing dissolved constituents of wood) removed from pulp as digester is dumped

Weak red liquor: Sulphite waste liquor (approximately 15% solids) which is further concentrated in cascade and multiple effect evaporators.

Strong red liquor: Sulphite waste liquor (60% solids), which has been through evaporators

Sulphur consumption: Pounds of sulphur (from elemental sulphur, pyrites, or SO_2) consumed per ton of air-dry pulp, determined from operating and inventory records.

Magnesium oxide consumption: Pounds of base material consumed per ton of air-dry pulp, determined from operating and inventory records

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About 1936, Tomlinson of Howard Smith Paper Mills, Ltd., found that, contrary to the generally accepted view, when a pure magnesium-base liquor is burned, magnesium sulphate did not reduce to the sulphide in the presence of carbon, but instead had the unique characteristic of reducing to the oxide with the liberation of sulphur dioxide. When the liquor is burned under certain furnace conditions, the oxide produced is exceedingly reactive in combining with sulphur dioxide. He also found that the magnesia-base liquor could be evaporated to high concentrations with comparative freedom from insoluble scale formation. These discoveries are the heart of the cyclic magnesia-base process of pulping wood by the sulphite method (*11*).

The first pilot plant to make pulp by the new cyclic process was installed as a joint venture by the Howard Smith Paper Mills, Ltd., and The Babcock & Wilcox Co., in 1937 at the Cornwall, Ont., mill of Howard Smith. In 1940 these companies announced the results of several years of joint experimental work and pilot plant operation of the magnesium oxide cyclic process (*12*). At about the same time, Weyerhaeuser Timber Co. was independently developing the same process, and a pilot plant was operated at the company's Longview, Wash., plant. A commercial digester was satisfactorily operated for about a year using magnesium-base acid, with recovery in a pilot plant furnace. The results of this pilot plant operation have been published (*2*).

Inasmuch as the three companies had common objectives in developing the magnesia-base process and in order to eliminate further duplication of effort, a cooperative arrangement was worked out by which all the experience gained in the several pilot plant operations and earlier research would be utilized in the conversion of a single commercial sulphite mill. An efficient and economical answer to the utilization of sulphite waste liquor could then be offered to the pulp industry. The Longview, Wash., mill of Weyerhaeuser Timber Co. was selected as the first mill to be converted to the magnesia-base process. It is one of the three branches of Weyerhaeuser's pulp division, which also operates a sulphite pulp mill at Everett, Wash., and sulphate pulp mills at Springfield, Ore., and Longview.

LONGVIEW, WASH., MILL OF WEYERHAEUSER TIMBER CO.

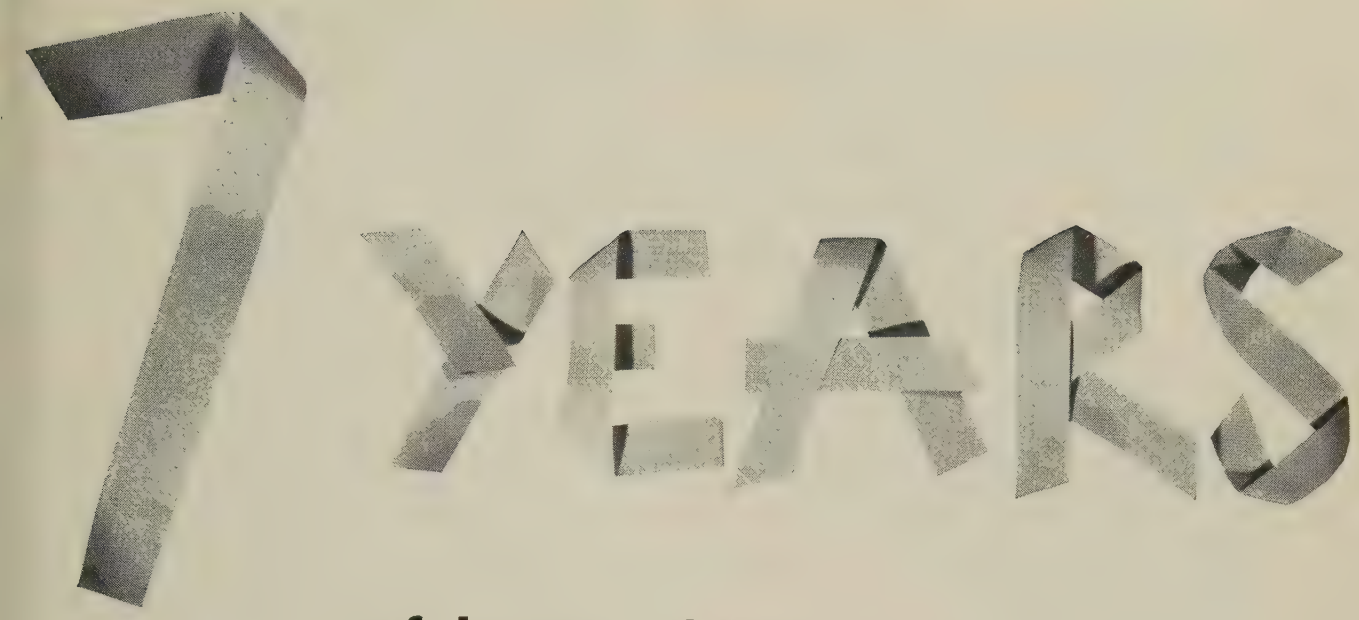
The sulphite pulp mill at Longview produces approximately 275 air-dry tons of pulp every 24 hours. Annual raw materials required for production of this output are shown in Table II. Pulpwood is chiefly hemlock (85%) and white fir (15%) which is grown at the company's St. Helens' tree farm in southern Washington, a part of Weyerhaeuser's 2,000,000 acres of timberland. Logs are brought by rail to Longview, where they are dumped into the Columbia River adjacent to the mill site for convenience in handling and

Table II. Approximate Annual Requirements, Weyerhaeuser's Sulphite Mill

Water, millions of gallons	6,432.4
Sulphur, tons	5,000.0
Magnesium oxide, tons	2,500.0
Chlorine, tons	4,600.9
Sodium hydroxide, tons	2,228.4
Lime, tons	855.7
Wood, air-dry tons	225,000.0

MgO sulphite and kraft mills at Longview, Wash.

Tall unit is acid fortification tower; center buildings are MgO recovery plant.



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sorting. Log ponds are provided for species of wood to be used in the various operations. A typical sawmill log haul lifts the logs from the water, and the logs are sawed to 24-foot lengths by a circular saw.

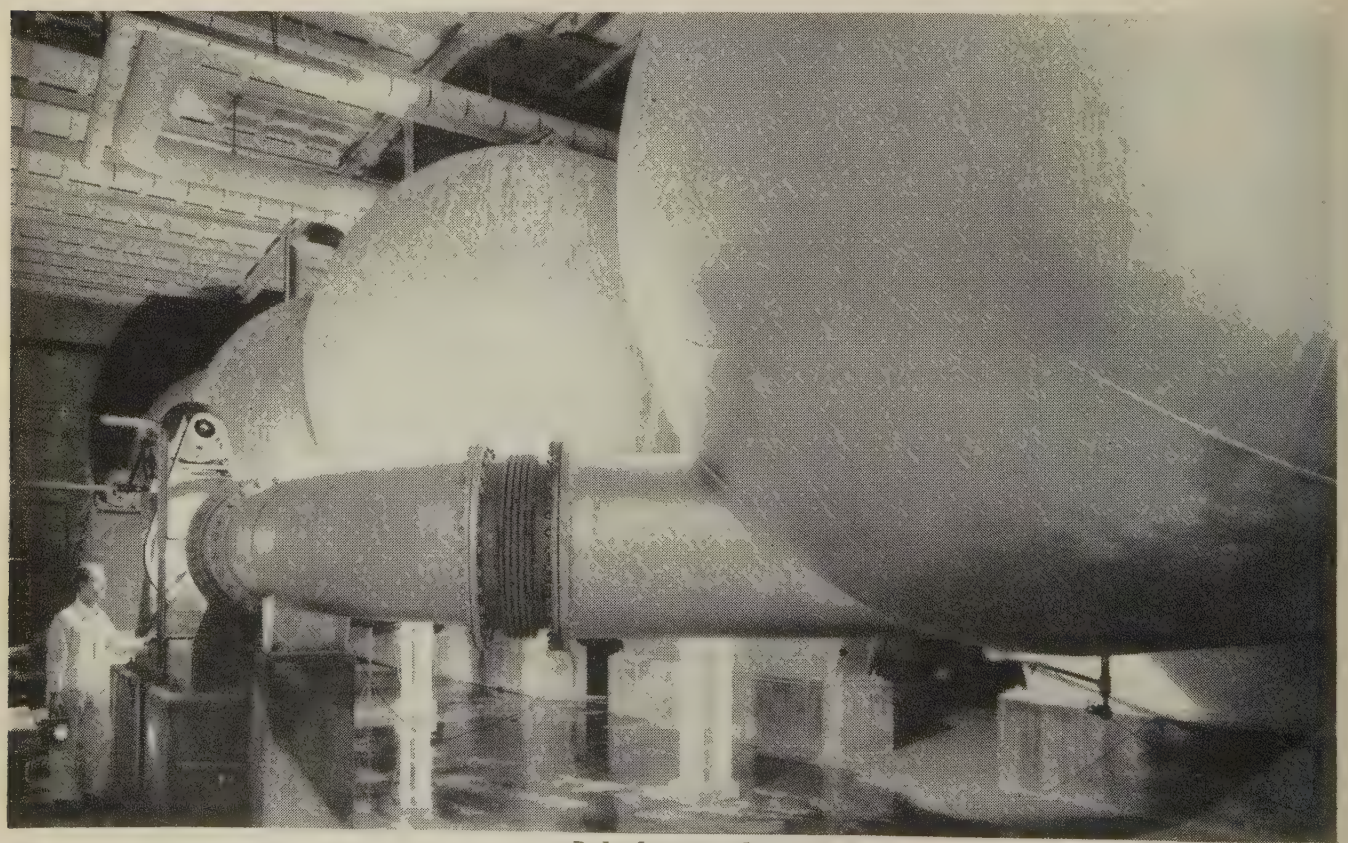
A chief requirement of quality in the production of pulp is that it be substantially free from dirt, particles of bark, and other objectionable matter. Thus logs must be completely cleaned of bark before processing. At Longview, this operation is performed by a hydraulic barker. The development of the hydraulic log barker during World War II by Weyerhaeuser introduced new economies in wood utilization and has now been adopted by many mills on the West Coast. The barker is of the chain-rotating type. As logs are rotated at a speed of approximately 4 r.p.m., water, at 1400 p.s.i., is applied through jets mounted on a traversing carriage moving at a maximum speed of 50 feet per second in the lengthwise direction of the log. Approximately 900 gallons of water per minute are used. After the log has been rotated one complete revolution, the barking nozzles are forced into a small end chamber out of contact with the log. The log-turning chain and supporting arms are lifted from the log, which is then ejected by an unloading mechanism. At this point, a new log is fed into the barker and the process repeated. One pass of the jet stream usually removes all the bark, but any that remains is removed by men called "spudders." Logs above 40 inches in diameter are reduced by band saws before further processing. Prewar barkers were broadly of two types—rotating knife barkers and drum barkers; the former is rapidly becoming obsolete. From the hydraulic barker, the bark is sent to a waste heat furnace, and the barked logs are ready for further processing.

In order that the cooking liquor may penetrate the wood quickly, completely, and uniformly, pulpwood must be reduced to chips for chemical pulp manufacture. This procedure is known as chipping and produces wood sections of uniform size, usually about $^{5}/_{8}$ to $^{3}/_{4}$ inch wide and $^{1}/_{8}$ inch in thickness. Pulpwood blocks from barking machines or storage which have been accepted as "clean" are fed directly to the chipper for this operation.

At Longview, logs are fed at a rate of 1 foot per second from a conveyor by sliding them lengthwise down an inclined chute and into the chipper spout. The wood is supported by a bed knife and chipped off as it comes into contact with four knives in a revolving steel disk, 170 inches in diameter and 7 inches thick; the disk is rotated by a 1500-hp., 240-r.p.m., synchronous motor. Average chip size is $^{13}/_{16}$ inch. In other installations, chippers with 8, 10, or 12 knives are in general use. Chips are screened for removal of slivers, knots, and fines and are conveyed by a rubber conveyor (30 inches wide and $^{3}/_{5}$ mile long) to a chip storage building which has a capacity of 1895 air-dry tons of chips equivalent to approximately 50 cooks.

The manufacture of pulp by magnesia-base pulping can be briefly summarized: chips prepared as described are cooked in an acid liquor. Lignin and other noncellulosic materials are dissolved during the cooking or digesting step by the active cooking chemicals—magnesium bisulphite and sulphur dioxide in aqueous solution. The nondissolved material is practically pure cellulose and is separated from the liquor on vacuum washers; after screening and bleaching it is subsequently processed into pulp sheets (Fig. 1).

The balance of the process is concerned with recovery



Pulp dump tanks

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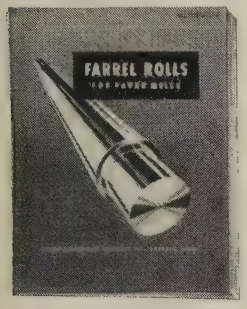

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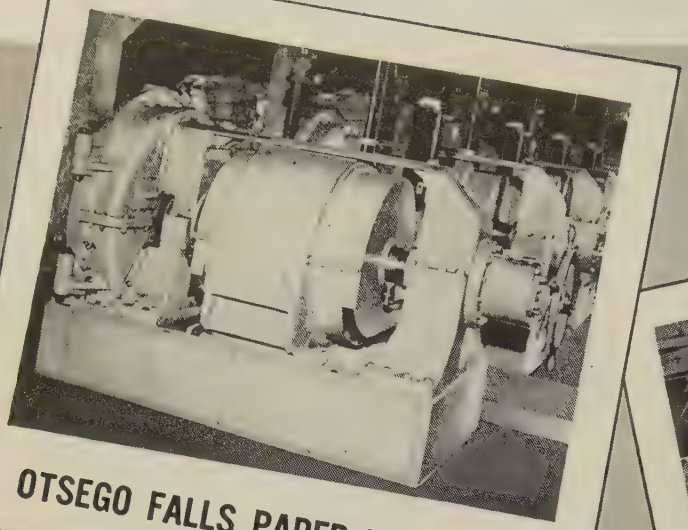

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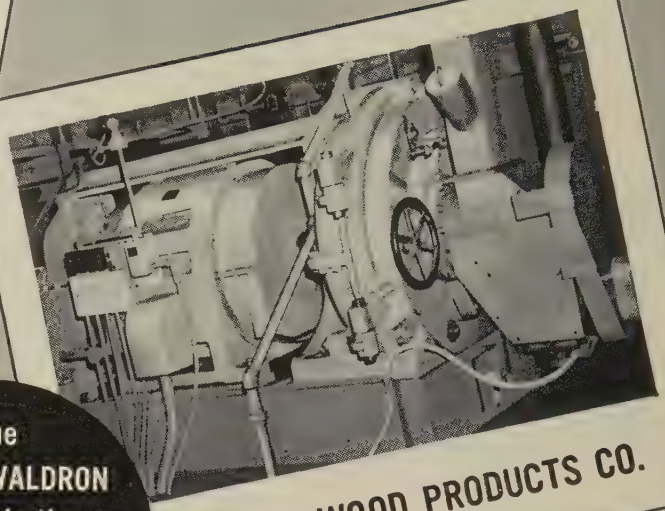

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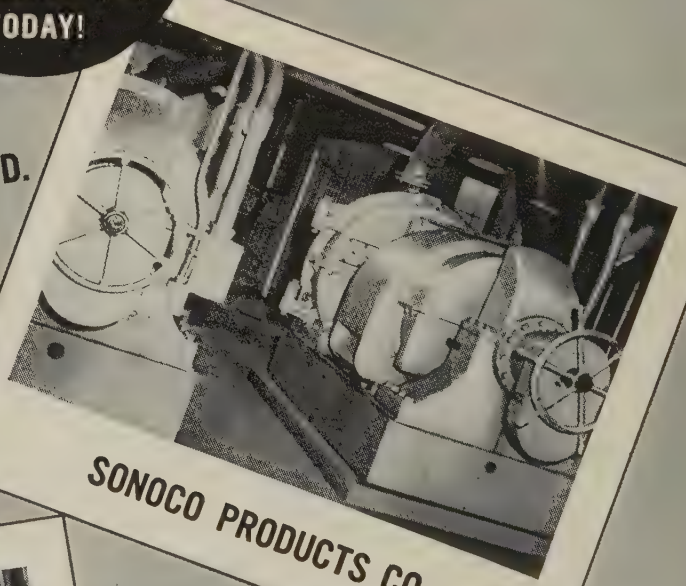

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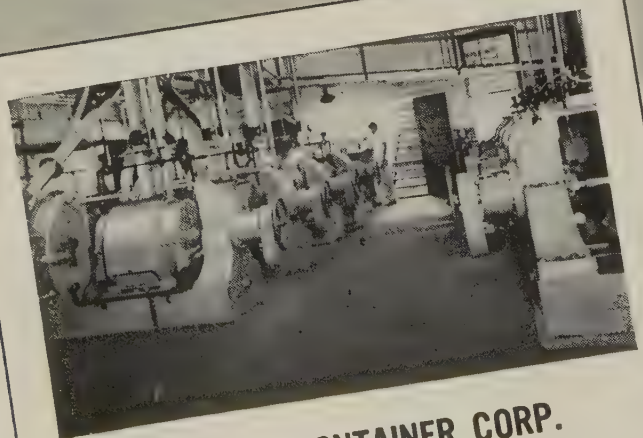

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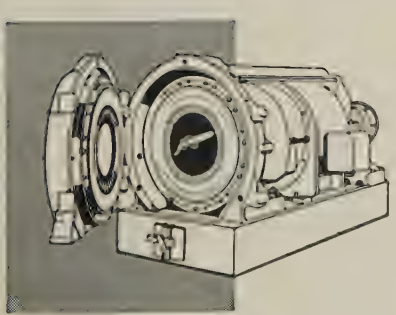

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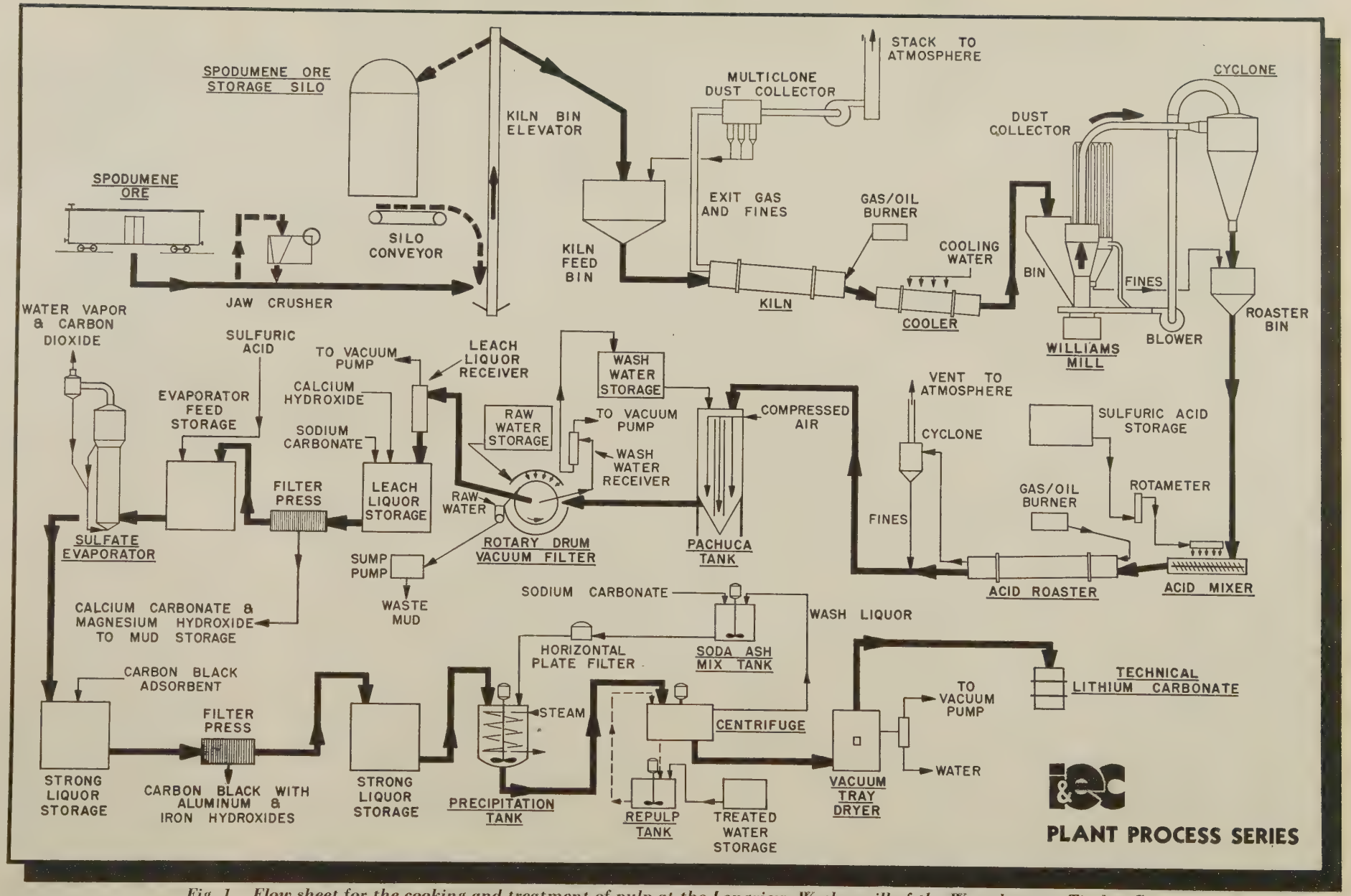
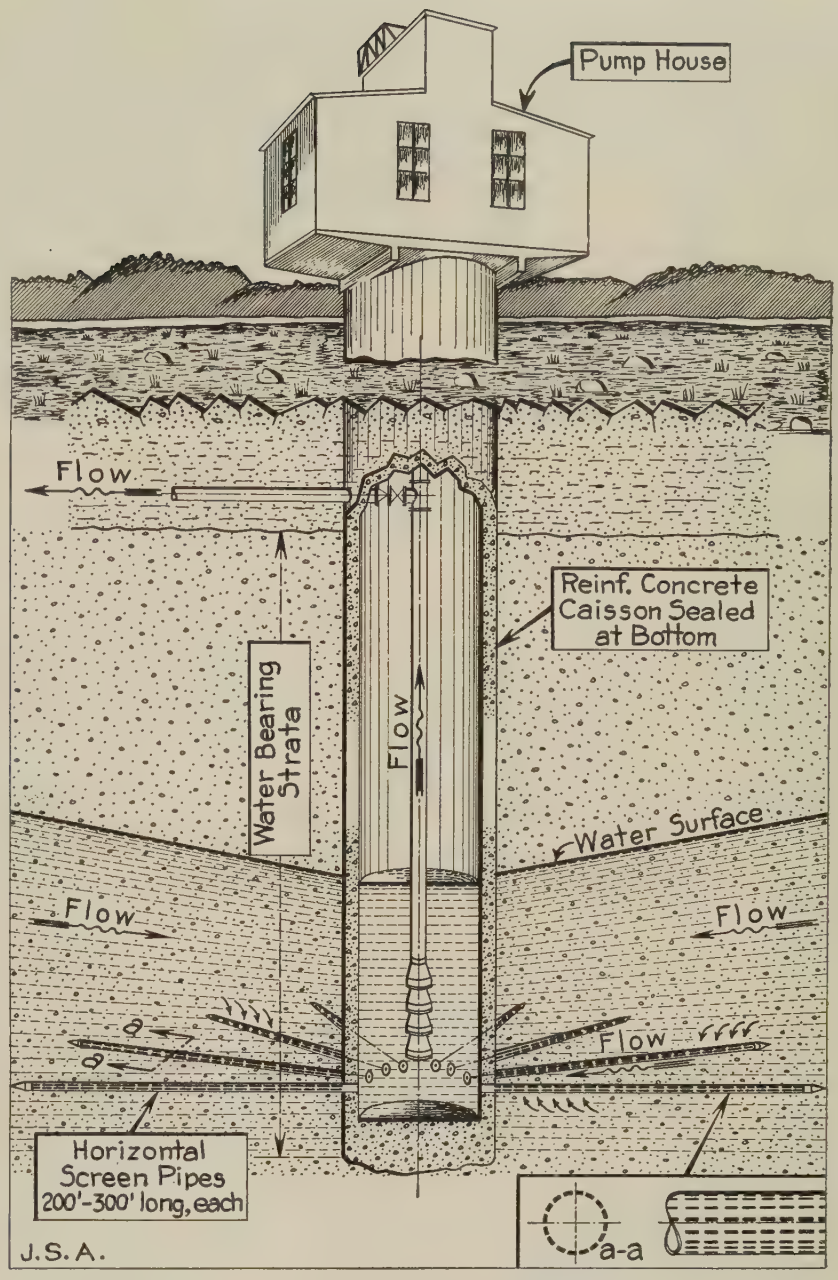


Fig. 1. Flow sheet for the cooking and treatment of pulp at the Longview, Wash., mill of the Weyerhaeuser Timber Co.

Cutaway view of a typical collection unit. The Ranney Process is simply one of purifying ground water by percolating it through sand and gravel.



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and re-use of the chemicals used in digesting the wood. The sulphite residual liquor or red liquor recovered as filtrate from vacuum washing the pulp is evaporated to 55 to 60% solids in multiple-effect evaporators and burned in a special furnace. In the incineration of the liquor, heat is created for the boilers and magnesium oxide and sulphur dioxide are formed and leave the furnace with the flue gas. The highly reactive magnesium oxide is collected in multiclones and conveyed to absorption towers, where it is re-used in making cooking liquor. Sulphur dioxide gas from the furnace is cooled and fed into the bottom of the towers; magnesium oxide, as a water slurry, is fed to the top of the towers. The acid is fortified to standard strength with make-up sulphur dioxide obtained from the burning sulphur.

Cooking

There are eight cookers or digesters at the Longview mill. These are large cylinders, 6400 cubic feet each, equipped with conical bottoms and bell tops. The digesters are fabricated from 1-inch mild steel plate and have a double acid-resisting brick lining; overall height is 52 feet and diameter is 16 feet. The rated capacity of each digester is 16 tons of air-dry pulp per cook. Each digester is equipped with an indirect steam heater to prevent dilution of the waste liquor with steam condensate. These single-pass tube heaters are made of type 316 stainless steel, with a total heating surface of 450 square feet per heater; steam is used at 125 p.s.i.g. and 410°F. Steam requirements for pulp digestion and other processing steps are shown in Table III.

Wood chips are carried by a belt conveyor to a 42-inch filling belt over the digesters. The filling hopper can be moved from digester to digester for charging. A charge consists of approximately 62 tons of chips (47.8% water), which is fed through the top of the digester. The head is then bolted down and pulp sheets are used as gasket material. The "hot acid" or Chemipulp process is used: acid at 65°C. is fed into the digester from the acid accumulator at a ratio of 4.0:1 of oven-dry wood charged. Total acid to a digester is approximately 21,000 gallons. Red liquor at 160°F. is added to fill the digester. Approximately 6000 to 7000 gallons are required. Cooking acid compositions at various points in the process are shown in Table IV.

Cooking liquor is circulated through the heater and digester by a centrifugal pump (4000 g.p.m.) driven directly by a 75-hp., 2200-r.p.m., 2200-volt motor. Collector rings or strainers, made of type 316 stainless steel, with $^{5}/_{16}$-inch holes spaced $^{19}/_{32}$ inch between centers, are located at the bottom of the cylindrical section of the digester. The circulating liquor passes

Table III. Steam Requirements

Use	*Steam used/ton air-dry pulp, lb.*	*Pressure p.s.i.*	*Temp., °F.*
Red liquor evaporation	3200	40	320
Pulp digestion	3200	125	416
MgO furnace operation	850	40	320
Bleach plant	1175	125	416
Pulp drier	3150	35	..
Wood barker	1000	125	416
Misc. (acid making, heating, auxiliary equipment)	100	125	416
Steam generated in waste heat boiler of MgO recovery furnace	7800	620	720

Table IV. Temperatures and Typical Cooking Acid Analyses

	Sulphur dioxide, %			
	Total	*Free*	*Combined*	*Temp., °C.*
Furnace gas absorption tower	4.60	2.30	2.30	31
Fortified raw acid	5.75	3.50	2.25	32
Storage tank acid	6.50	4.50	2.00	36
Low pressure accumulator	8.40	6.50	1.90	50
High pressure accumulator	8.60	7.00	1.60	65

through the strainers and heaters, and then the flow is divided; 25% goes to the bottom digester fitting and the remainder, through a header ring, to three spray nozzles at a point near the top of the digester.

At the end of 3.5 hours, the temperature reaches 139 to 140°C., and the pressure is approximately 90 p.s.i. Steam to the circulating system is cut off just before the maximum temperature is reached. At this point, cooking liquor is relieved by side relief to the accumulator to a level of 13 feet from the top of the digester. This decreases the quantity of liquor that must be removed from the pulp later and provides gas space for later "gassing off" or relief of sulphur dioxide and steam to the acid-fortifying system. At the end of 7 hours the circulation of the cooking liquor is stopped and digestion is almost completed.

Dumping and Washing

A departure from the conventional practice of blowing a digester into pits is practiced in this particular magnesium oxide plant. When the cook is completed, the pressure is relieved to near atmospheric. This is done in two steps: from 90 to 35 p.s.i., the pressure is relieved into the accumulator; from 35 p.s.i. to as near as possible to atmospheric, it is relieved to the acid storage tanks after being passed through a single-pass, indirect heat exchanger. An eductor is used to shorten the time required to relieve the digester to zero pressure, and approximately 20 minutes are required for this additional relief. The digesters are connected to a common header and the stock is flushed out by back-filling with red liquor and pumping. Red liquor is pumped to the suction side of the digester circulating pump and can be diverted to the bottom, top, or collection ring section of the digester. In the flushing or dumping step, the pulp is diluted to approximately 3.5% air-dry consistency. A trap, commonly called a "brick catcher" is in the flow line between the digesters and the dump tank. The brick catcher removes any particles which the pulp may have picked up from the lining of the digesters.

The 70-foot steel dump tanks, which are connected and used in pairs, are of unusual design and novel appearance. The tanks are set at a slope of $^{7}/_{32}$ inch per foot, the low end being 3 feet above floor level. Each pair of these tanks provides capacity of 183,000 gallons; circulation is provided by 500-r.p.m. pumps operated by 20-hp., 550-volt motors with V-belt drive. The pulp is pumped from the dump tanks by a centrifugal pump (2300 gallons per minute) operated by a 40-hp., 550-volt, 1800-r.p.m. motor.

In the dumping step, only 5 to 10 pounds of sulphur per ton of pulp flash off as sulphur dioxide gas. Even though the pressure at the time of dumping is very nearly zero, the static head on the digester prevents

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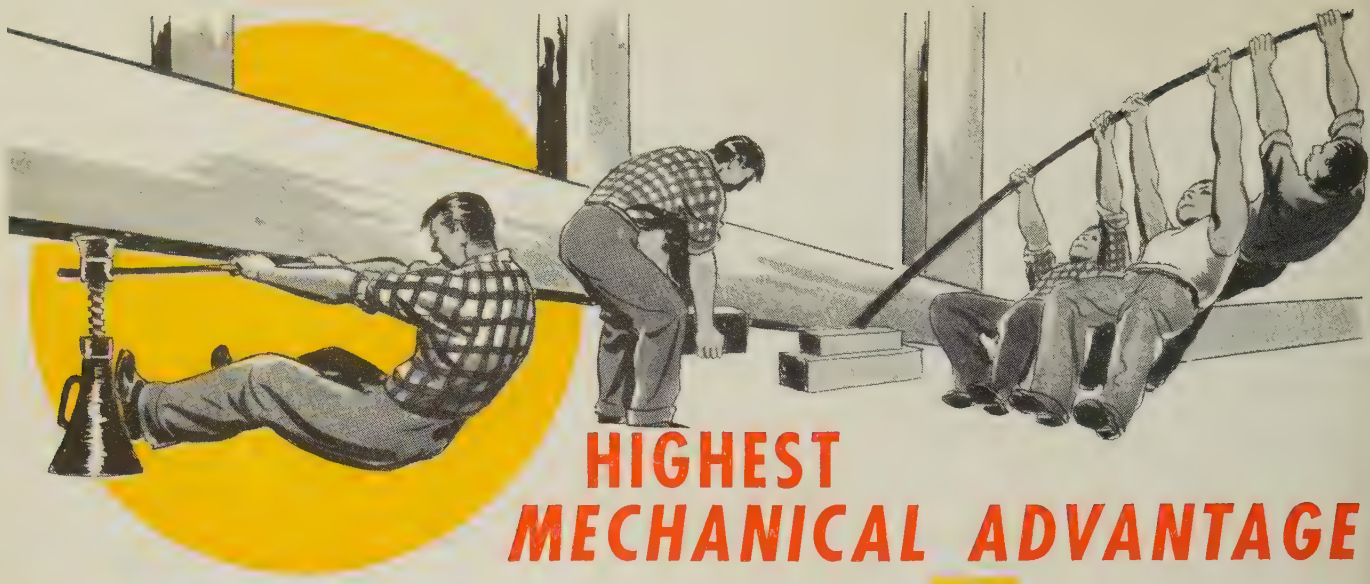

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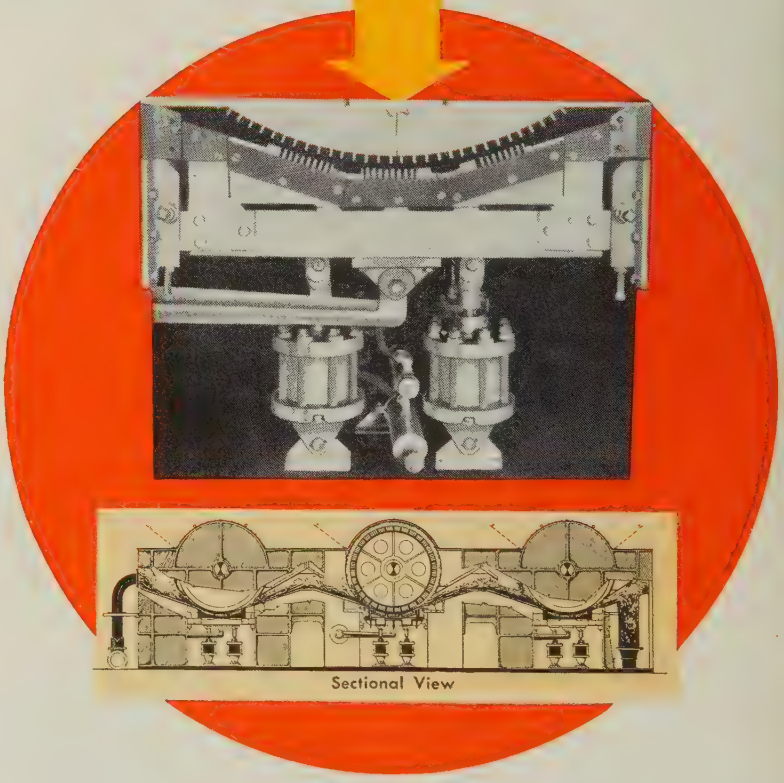

Sectional View

To our sales department, that definition means the efficiency, economy and high production for which the *Controlled Flow* VICTORY BEATER* is famous. To you it means more and better stock refining at a lower cost . . . often the difference between profit and loss, success and failure.

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Moreover, Nopco 2252-A has proved exceptionally satisfactory as a beater size in conjunction with rosin in the proper balance.

Yes, here indeed is a "double-duty" size—one highly efficient for surface sizing yet equally useful for internal application.

You'll want to profit by trying Nopco 2252-A now. Our technical representative will gladly give you full information or arrange for a supervised run in your own mill soon. Invite him to discuss Nopco 2252-A the next time he calls, or write.

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relieving all the free sulphur dioxide. Also, it is not possible to obtain good circulation at the latter part of the cooking cycle. This sulphur dioxide is recovered by venting the dump tanks to the furnace flue gas absorption system.

Some of the early proposals for the magnesium oxide process indicated that ratios as low as 2.5 pounds of total liquid per pound of oven-dry wood could be used satisfactorily (*6*). At the present time, the ratio has been reduced from 5.1:1 (calcium-base pulping) to 4.0:1 and the reduction thus far saved 6000 to 7000 gallons of cooking acid per digester charge. Ratios between 2.5:1 and 3.5:1 are still considered as practical for future operations, and additional savings in cooking liquor and consequently steam for cooking and evaporation will be realized.

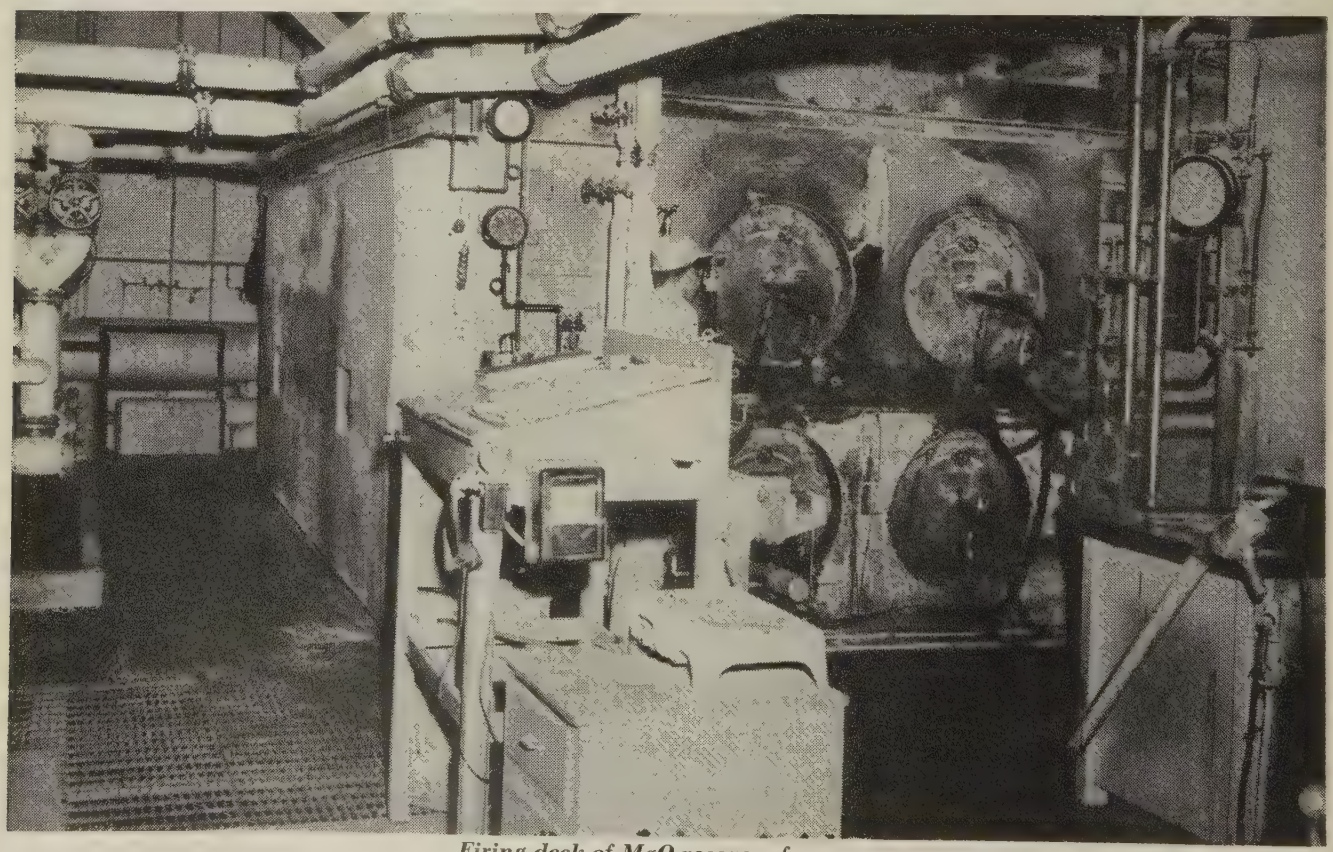
Prior to washing, the pulp is freed of knots by four 3 by 4-foot vibrating knotters operated in parallel. Screens are constructed of $^3/_{16}$-inch type 316 stainless steel and openings are $^3/_8$ inch in diameter. The stock is assisted through the screen plate by vibration of the screen frame. Oversize material, knots, etc., retained by the screen are carried to the discharge end of the screen frame and dropped into a tailings trough. No attempt is made to recover the residual waste liquor in the knots since the quantity is small. Knots and oversize particles of pulp are defibered and mixed with screenings and sold for boxboard manufacture.

Material passing through the $^3/_8$-inch openings is ready for washing and filtering and passes by gravity to a three-stage vacuum washer. Three 8 by 16-foot stainless steel vacuum washers, set in concrete vats with acid-resistant tile linings, are used. The piping arrangements and liquor circulating systems are similar to a conventional brown stock washing system

Table V. Longview Mill Production Control Data
(All Tests by TAPPI Standards or Modifications)

	Pulp grade		
	Paper-making	*Nitrating*	*Absorbent*
Per cent of total production	80	10	10
Chemical testing of pulp, %			
Alpha-cellulose	90.2	92.9	91.2
Beta-cellulose	1.4	1.9	2.4
Gamma-cellulose	8.4	5.2	6.4
Ash	0.15	0.12	0.09
Fe	0.0003	0.0004	0.0004
Cu	0.0005	0.0005	0.0006
Ca	0.09	0.06	0.06
Si	0.006	0.006	0.005
Ether-soluble extractives	0.35	0.32	0.28
Alcohol-soluble extractives	0.04	0.02	0.04
Total pitch	0.39	0.34	0.32
Copper No., Schwalbe-Haglund method	1.5	1.2	1.6
10 % KOH solubility, %	13.3	11.6	15.8
Viscosity, centipoises	247	135	134
Physical testing of sheets			
Brightness, % on G.E. instrument	88.0	88.0	88.0
Bursting strength (Mullen), p.s.i./unit wt. × 100	135	110	120
Aggregate tear, Elmendorf units/basic weight	1.6	1.9	1.6
Fold endurance	600	400	500

normally used in a kraft mill. The effluent from the third stage is pumped and sprayed on the pulp mat of the second stage; the effluent from the second stage is sprayed on the first-stage pulp mat. Temperature is maintained at approximately 180°F.; vacuum on drums is 3 inches of mercury. The filtrate from the washer, containing about 14 to 15% solids, goes to the weak red liquor storage tank for subsequent evaporation. The washed pulp at approximately 3% air-dry consistency goes to the unbleached pulp storage tank. This is a



Firing deck of MgO recovery furnace

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Carefully processed from a huge and exceptionally pure deposit of diatomaceous silica, Celite products will continue to meet the strict standards of quality established by Johns-Manville in the past.

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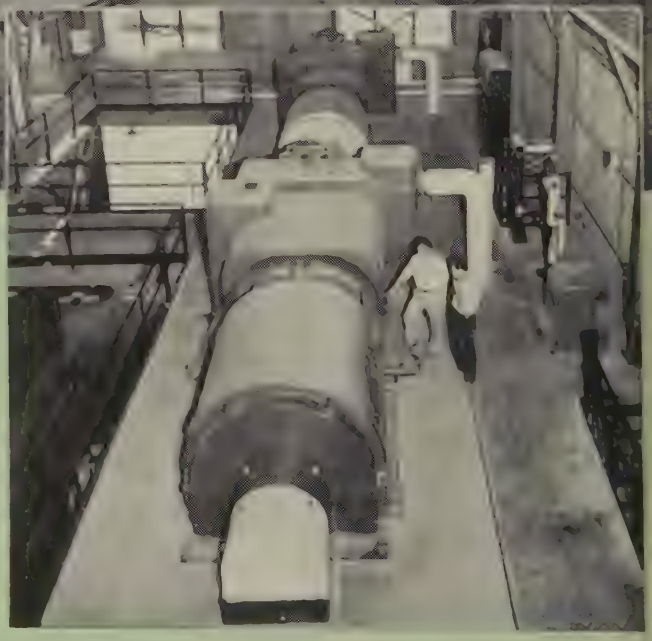

TWO POWERHOUSE STACKS mark Weyerhaeuser's new Pulp and Paper Mill at Springfield, Oregon. Leftover scraps from the Weyerhaeuser lumber mill at right are converted into Kraft Paperboard. Lumber is fully utilized in this operation. So is power. Westinghouse Specialists helped co-ordinate the electrical apparatus into a plant that makes full use of every kilowatt generated.

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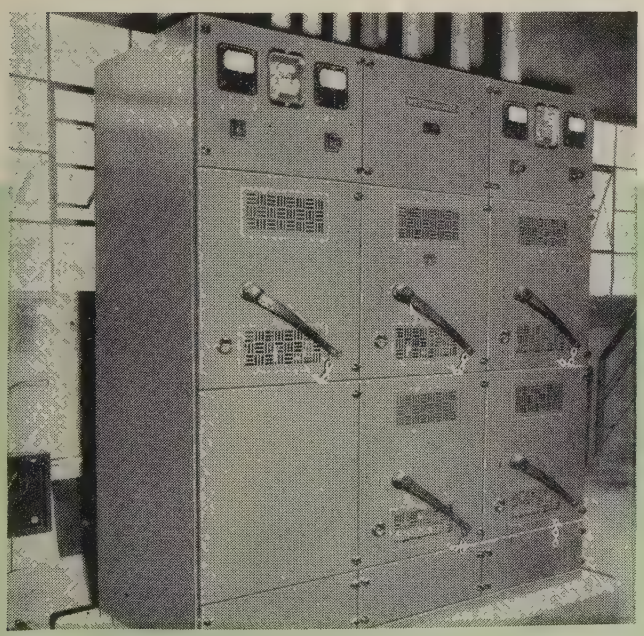

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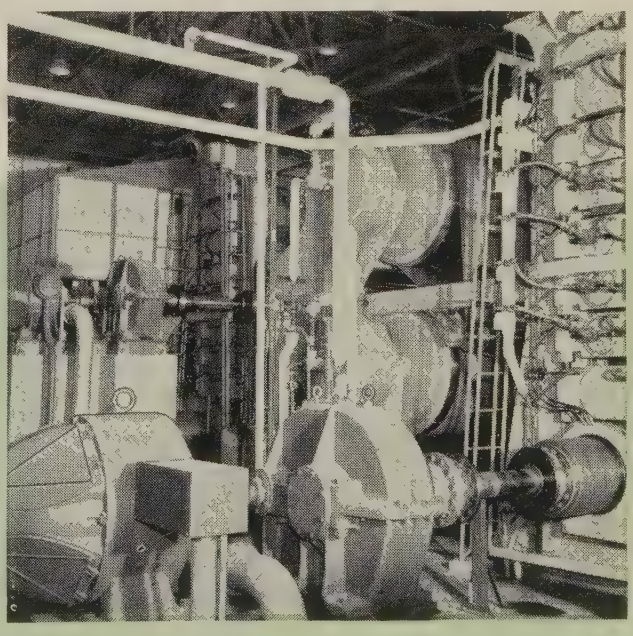

UTILIZATION . . . The mill's paper machine has a modern Sectional Motor Drive. Shown here—d-c motors driving the first and second calender stacks through Westinghouse Speed Reducers. Each motor is supplied by its own d-c generator. Hence, each section of the paper machine can be separately, accurately controlled.

140,000-gallon wood stave tank, 36 feet in diameter and 18.5 feet deep.

A foam tank and foam breaker were installed in anticipation of foam occuring as in kraft pulp washing. However, very little foaming has been encountered, and at no time has foam carried over from the washer foot tanks. There are five additional stock tanks maintained for standby use and storage. These are similar to the unbleached pulp tank. A 282,000-gallon cylindrical wood tank is also used for standby and as a mixing tank when needed.

The pulp is further processed into one of three grades

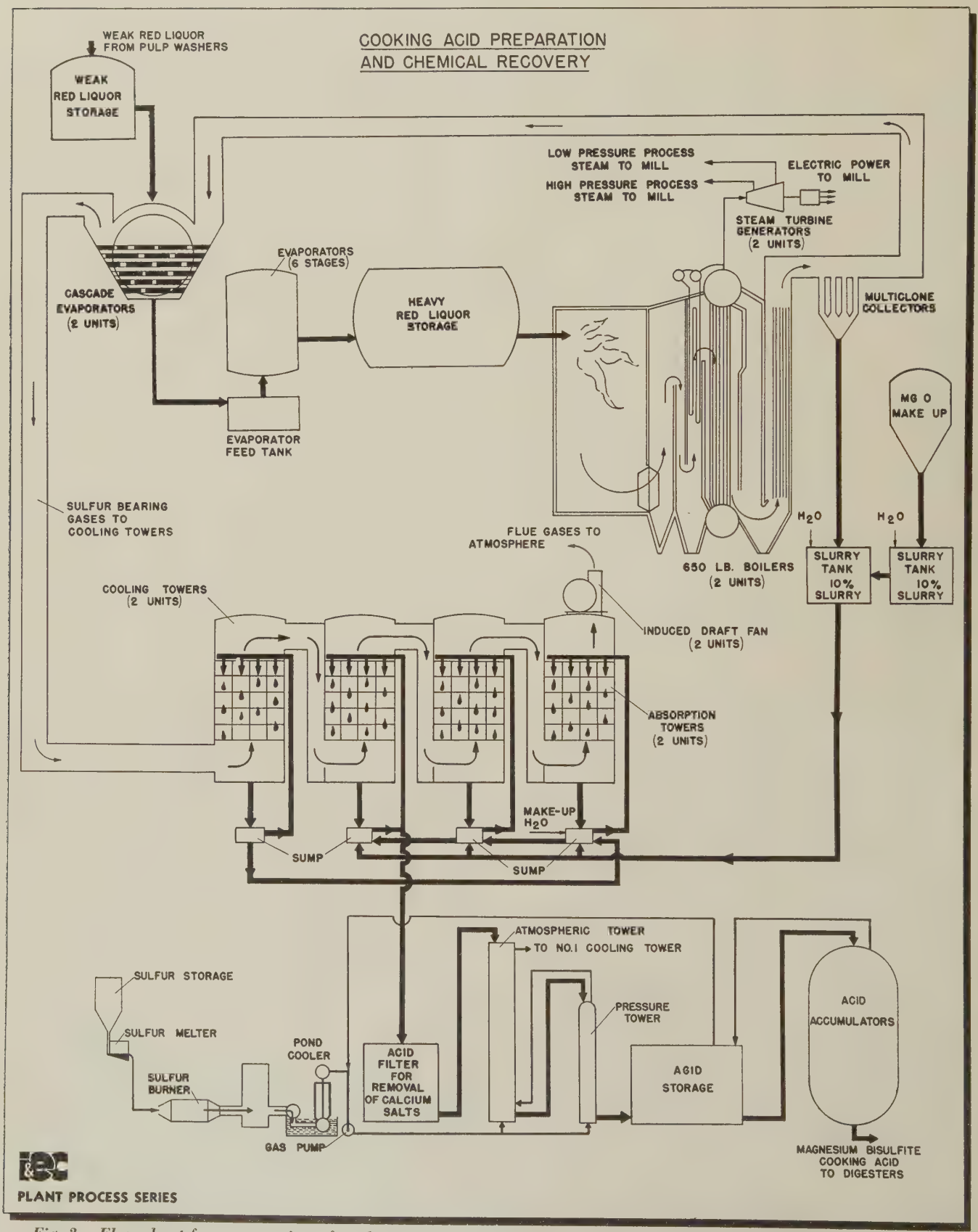

Fig. 2. Flow sheet for preparation of cooking acid and recovery of chemicals in magnesia-base pulping process

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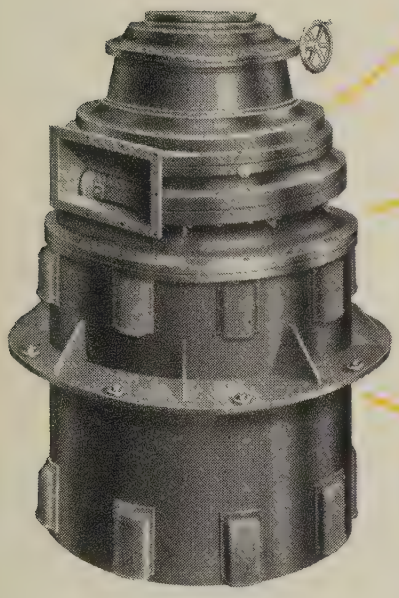

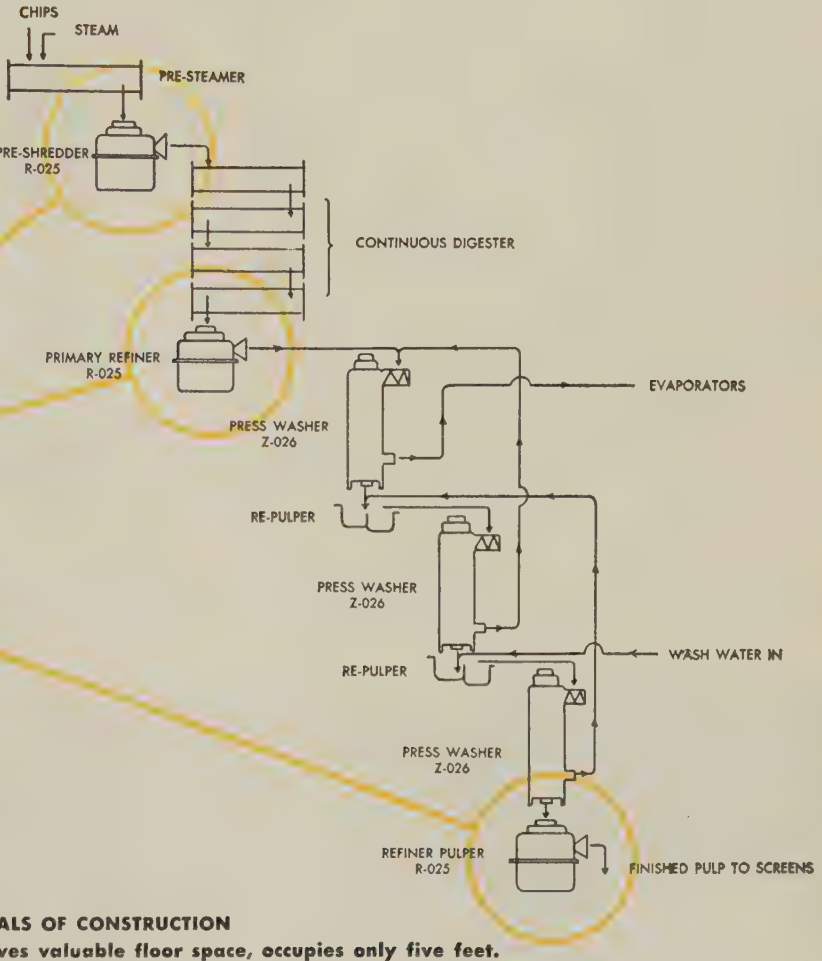

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Saves up to 50% of power normally required at low density.

Eliminates pumps which consume power and require expensive piping.

Reduces size and cost of chests.

Delivers a pulp with superior uniformity of fibre length.

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of finished pulp product as shown in Fig. 1; the properties of these pulps are listed in Table V.

Evaporation

Before the weak red liquor can be ignited and burned in the recovery furnace, with subsequent recovery of cooking chemicals, it must be freed of a part of its water content. This is done in two stages: It first passes through two cascade evaporators, which increase the oven-dry consistency from 15 to 17%. These cascades, of the single-wheel type, are 12 feet in diameter with 8-foot 7-inch faces. They are located in the furnace gas stream between the multiclone separators, where magnesium oxide is recovered, and the gas cooling towers (Fig. 2). Originally, the red liquor was first passed through the multiple-effect evaporators and then to the cascade evaporators for further concentration and consequent cooling of the recovery furnace gases before being fired in the recovery furnaces. However, this cycle resulted in the formation of foam and calcium sulphate scale in the cascades and caused interruptions in the fuel supply to the recovery furnaces. To prevent these occurrences, the liquor cycle was changed to pass the weak liquor from the washers through the cascades for initial concentration and finally to the multiple-effect evaporators.

In the multiple-effect evaporators, the weak red liquor is concentrated to approximately 60% solids. A six-body quintuple-effect evaporator is used. Outside heat exchangers and forced circulation are used to provide high tube velocity and to cut down on heating surfaces. Stainless steel or stainless steel-clad construction was used throughout the evaporation unit. Tubes are 1 inch in diameter and 12 feet long. Each heater has 1460 tubes or 4000 square feet of area. Red liquor enters the evaporators at 168°F. and leaves at 250°F.

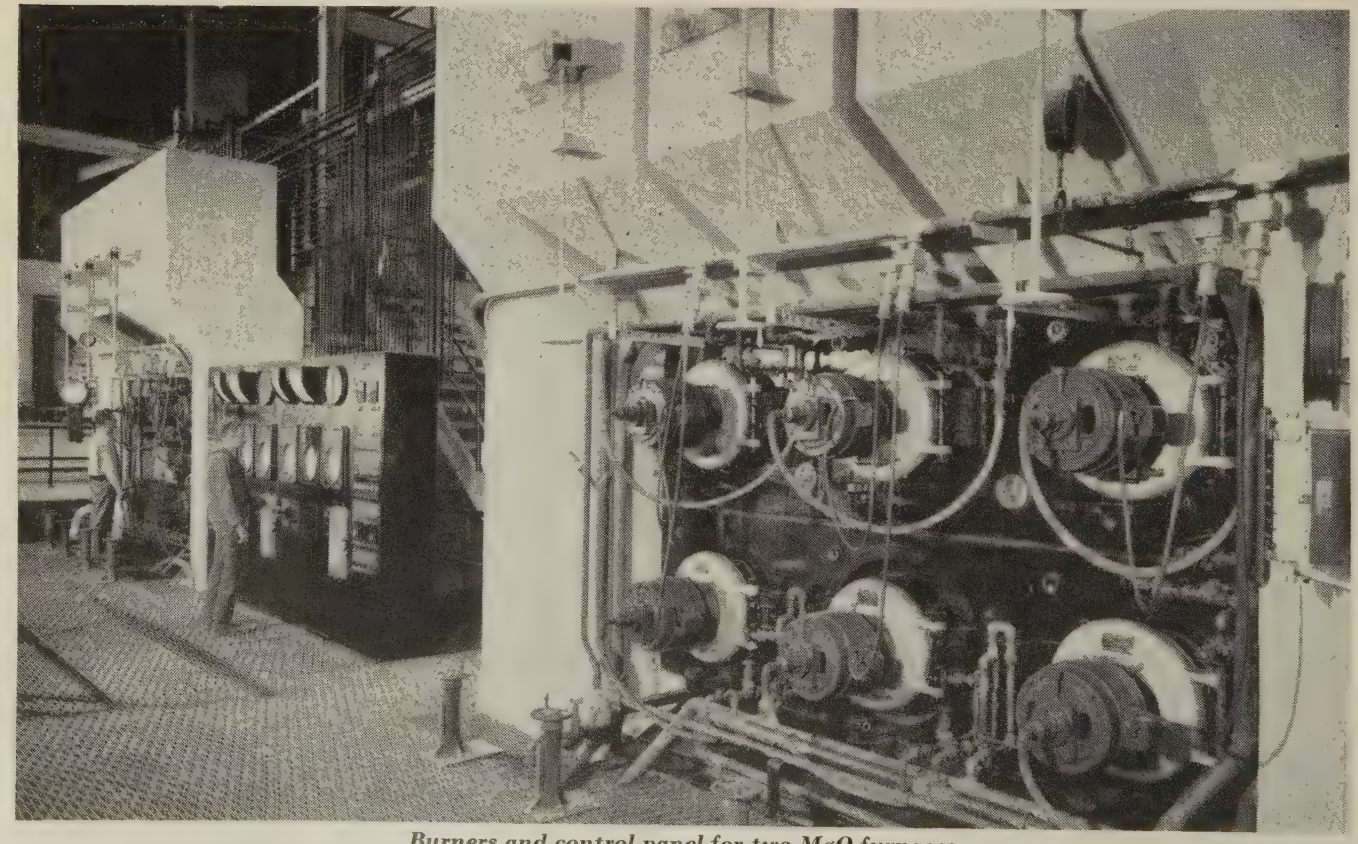
The evaporators are designed to produce 4.58 pounds of evaporation per pound of steam. Pounds of water evaporated per pound of steam used has actually been 3.25 because the unit is being operated temporarily as a quadruple effect with three first effect bodies for greater capacity. Weak red liquor is introduced into No. 2 effect, and the flow is concurrent with the vapors through Nos. 2, 3, and 4. The liquor then is transferred through 1C, 1B, and 1A.

A spare heating element and circulating pump are provided since it was anticipated that some difficulties would be experienced with scaling of heating surfaces. Scale formation was aggravated by abnormally high magnesium sulphate formation during preliminary operations when the rate of burning of liquor and sulphur was such that high quantities of excess air were admitted to the acid plant absorption system. During more stable operation, however, the quantity of sulphate formed is more uniform and of a smaller amount, and it is possible to operate the evaporator unit consistently at system rate.

The concentrated or strong red liquor goes to a storage tank and is ready for burning in the specially designed magnesium oxide recovery furnace.

Burning

Two furnaces used for the actual burning and chemical recovery in the magnesia-base pulping cycle are each attached to boilers of the two-drum Stirling extended furnace type, equipped with gas air heaters and rated as 137.5-ton recovery units. These units are rectangular in shape, $50^1/_2$ feet long by $18^1/_2$ feet wide and 38 feet in height. Each is designed for the produc-



Burners and control panel for two MgO furnaces

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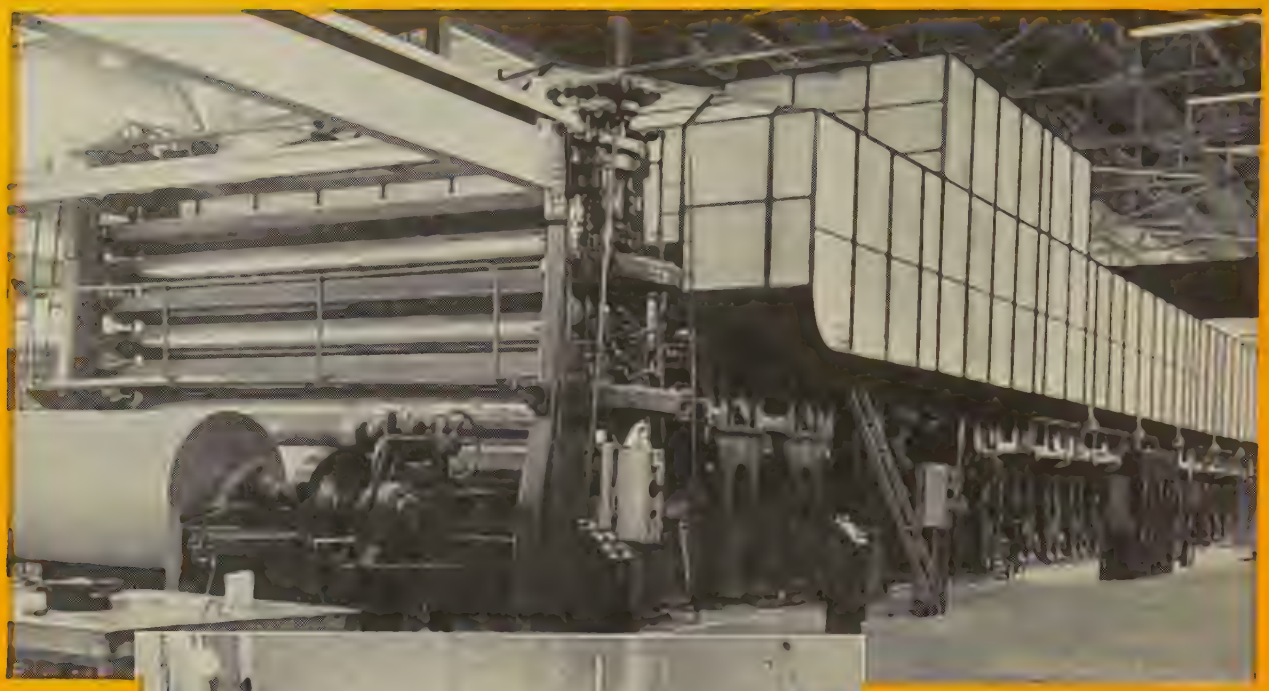

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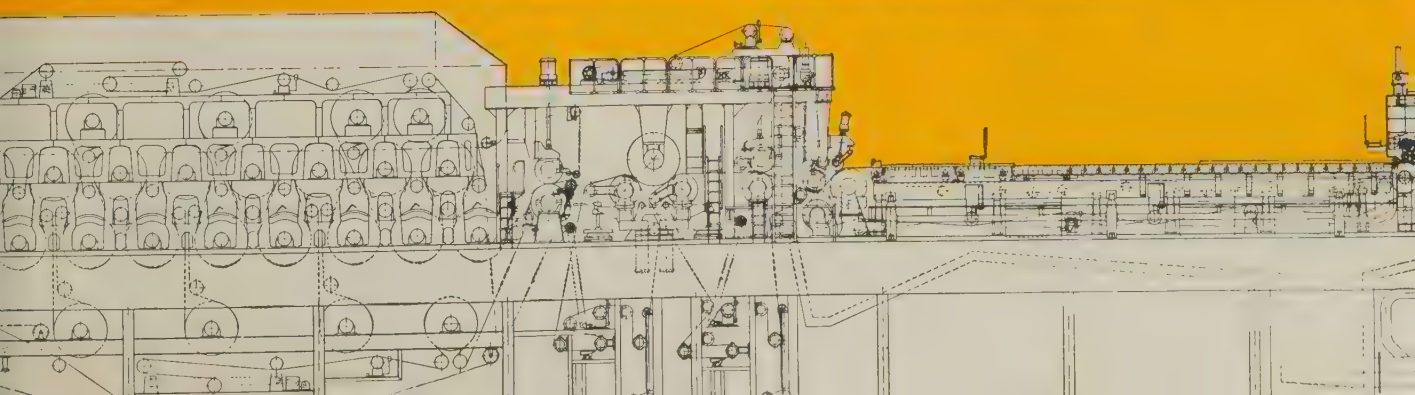
Other new features include controlled tension on the drinier wire, improved va connections to the flat boxes quick handling device for re ing and replacing individual tion boxes. Air loading has incorporated in the calender and the reel redesigned to vent vibration and jumpin the end that "bull's eye" rol produced.

8-roll Calender stack, Pope and a high speed Winder special roll handling devic provided at the finishing The machine is driven by electric sectional drive wit dividual section generators full electronic controls.



tion of 69,600 pounds of steam per hour at 615 p.s.i. and 725°F. The strong or heavy red liquor is fed into the furnace, at the second floor level, through four steam atomizing Y-jet burners on each side of the furnace and forced draft air at 750°F. for combustion is introduced through the double casing around the burners. The concentrated red liquor is atomized, into the furnace, at 60 to 65 p.s.i. and 235°F. through the stationary nozzles. The actual combustion chamber or fire box is approximately 13 feet wide by 14 feet deep by 18 feet high. The concentrated sulphite liquor is burned under self-sustaining combustion conditions with furnace temperature and gas velocity producing a dry magnesium oxide ash of high purity and reactivity. Separation of the magnesium oxide ash from the furnace gases is accomplished by the use of multiclone separators located after the air heater and preceding the cascade evaporators (Fig. 2). The multiclones are equipped with star valves at the bottom discharge permitting operation against the induced draft. The magnesium oxide ash is conveyed by a screw conveyer to a slurry tank where make-up magnesium oxide is added and the slurry pumped to the sulphur dioxide absorption towers. The furnace gases from which the ash was removed go to the cascade evaporators previously described and then to cooling towers before passing finally to the sulphur dioxide absorption towers.

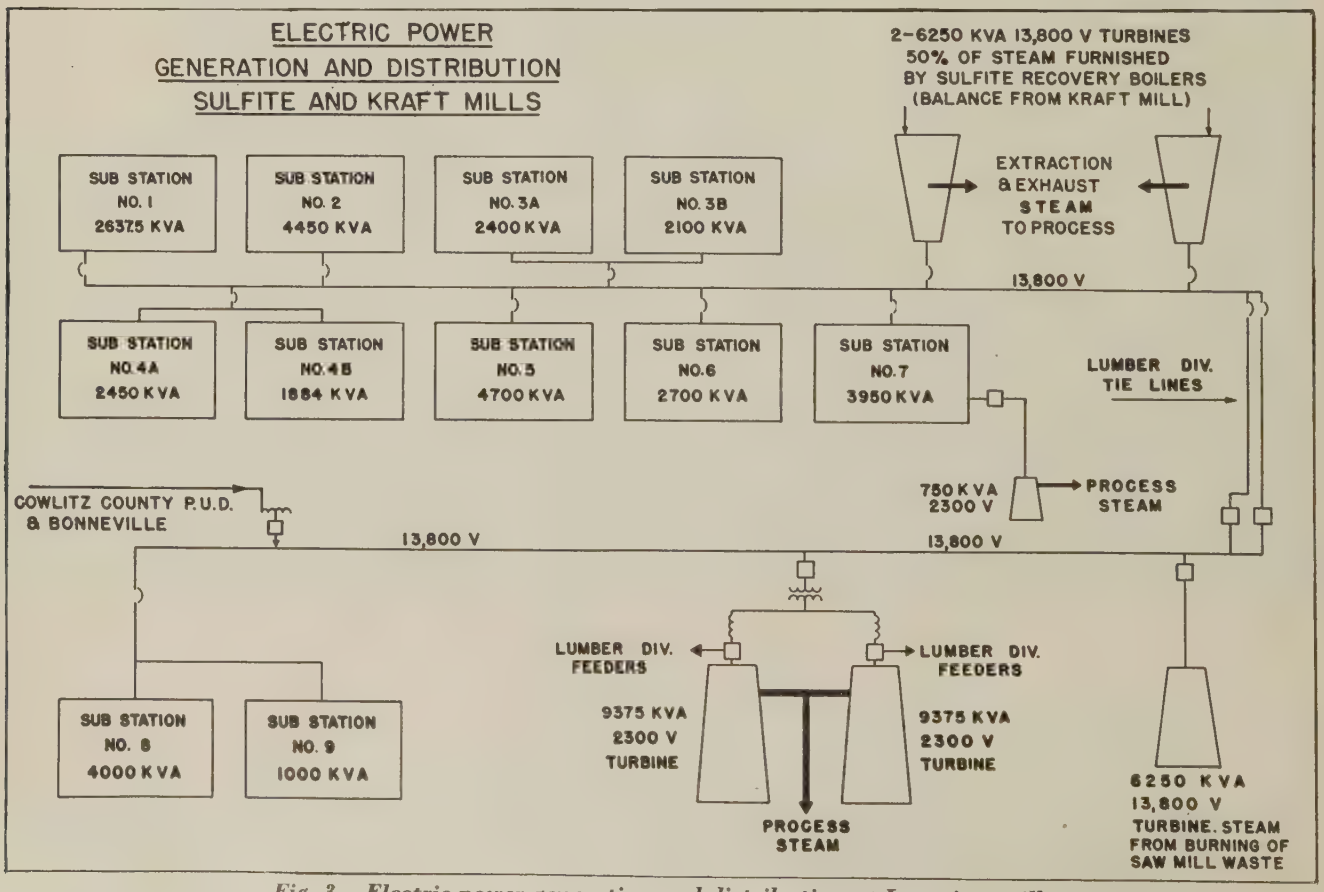
The boiler plant produces steam for two 6250-kva. turbo-generators in an adjoining room where power for operating both the sulphite and sulphate mills is generated (Fig. 3). The generators operate on steam at 725°F. and 615 p.s.i. The turbines exhaust at 35 p.s.i. and bleed at 135 p.s.i. The power plant is fully and automatically controlled from a central control board. Power distribution is shown in Fig. 3.

In pilot plant operations and the first installation at Longview, mechanically driven integral fan oil burners were used. However, considerable difficulty was experienced with baking of organic solids on the inner cup of the burner under continuous operating conditions. The steam atomizing burners were installed to overcome this difficulty, and since then it has been possible to operate the units at expected capacity with the production of reactive ash of very good quality. Some difficulty has been encountered with fouling of boiler passes or tube surfaces, and hand lancing is sometimes necessary to keep the furnaces relatively clean and in condition for continuous operation. To facilitate cleaning, automatic soot blowers have been installed.

Acid Making

To complete the cyclic process, the cooking liquor, as it is used in digesting the wood chips, must be prepared from the recovered chemicals. This part of the process is shown in Fig. 2.

The sulphur dioxide gas from the furnaces at 300°F. is cooled to 120°F. in two all-steel cooling towers lined with acid-resisting brick and filled with 4 and 6-inch ceramic partition tile. The towers are 16 feet in diameter and 50 feet high. The circulating cooling water containing some sulphur dioxide is cooled from 160 to 110°F. with twelve sections of water-cooled heat exchangers and then returned to the top of the gas cooling tower. The cooled sulphur dioxide gas is then absorbed in six concrete absorption towers in two series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STEAM
TO PROCESS
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SUB STATION NO. 3A 2400 KVA
SUB STATION NO. 3B 2100 KVA
13,800 V
SUB STATION NO. 4A 2450 KVA
SUB STATION NO. 4B 1884 KVA
SUB STATION NO. 5 4700 KVA
SUB STATION NO. 6 2700 KVA
SUB STATION NO. 7 3950 KVA
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PROCESS STEAM
COWLITZ COUNTY P.U.D. & BONNEVILLE
13,800 V
13,800 V
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9375 KVA 2300 V TURBINE
9375 KVA 2300 V TURBINE
SUB STATION NO. 8 4000 KVA
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6250 KVA 13,800 V TURBINE. STEAM FROM BURNING OF SAW MILL WASTE

Fig. 3. Electric power generation and distribution at Longview mill

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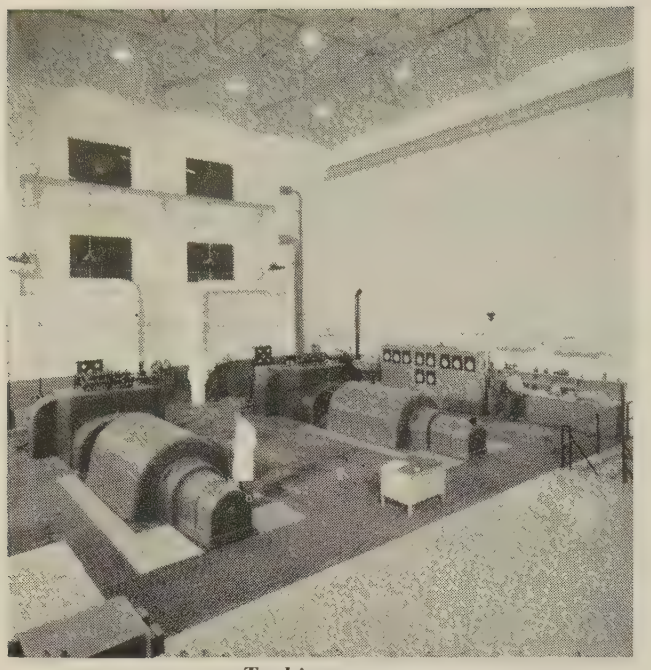
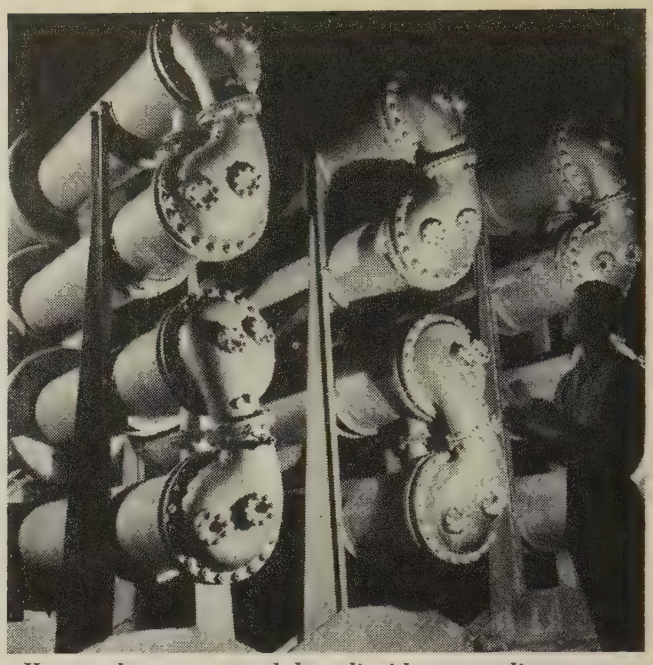


Turbine room

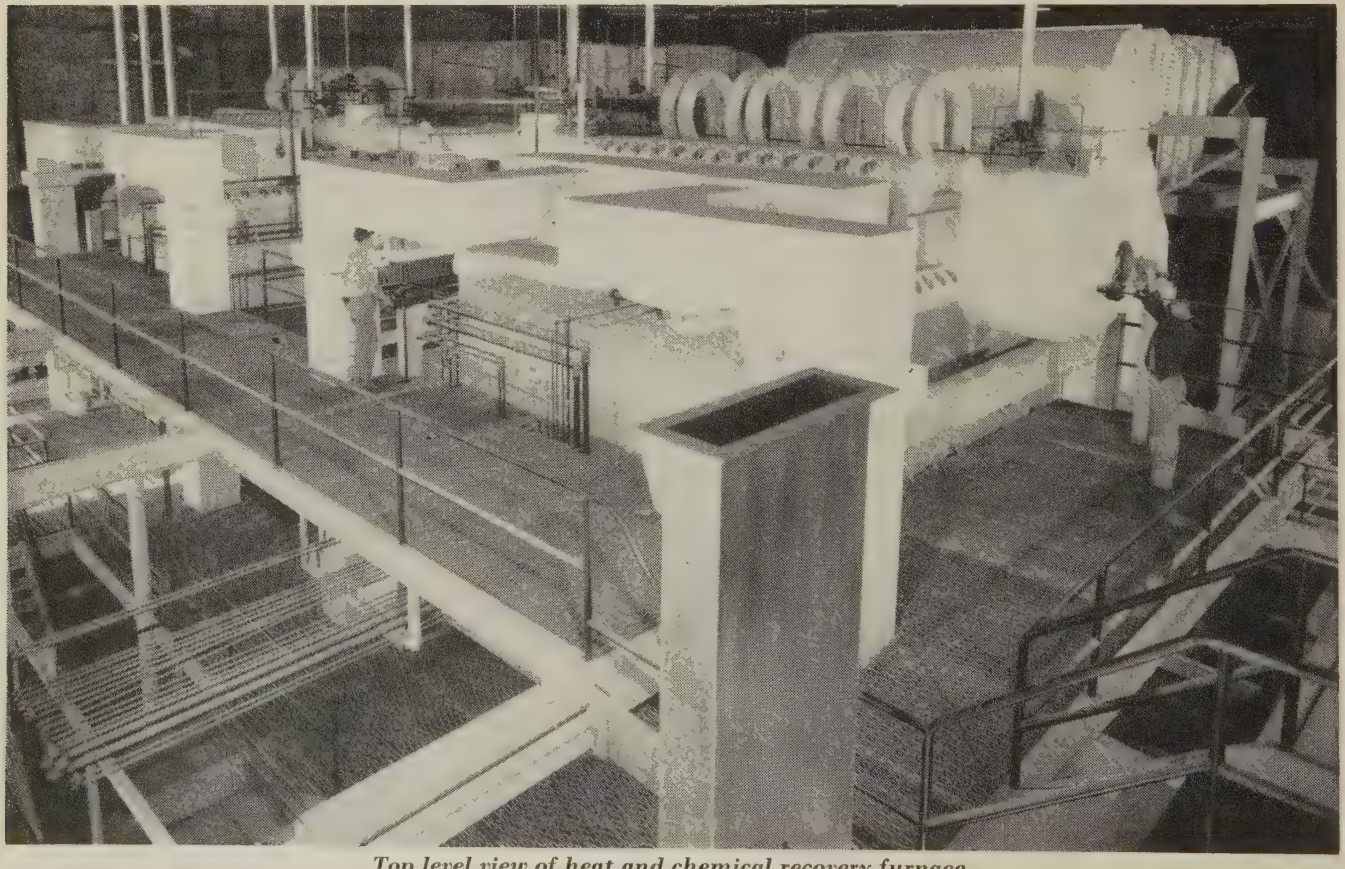
Two noncondensing turbines and 6250-kv.a. generators.



Heat exchangers on sulphur dioxide gas cooling towers

of three each following each of the two recovery units. The towers are 16 feet in diameter and 50 feet high, have a 3-inch acid-resisting tile lining, and are packed with 6-inch cross partition ceramic tile supported by carbon uprights and beams. The towers are necessarily large since the concentration of sulphur dioxide is of the order of 1% or less, and the quantity to be absorbed is approximately 360 to 400 pounds of sulphur dioxide per ton of pulp produced.

A 10% magnesium hydroxide slurry, from the magnesium oxide slurry tank, is fed at the rate of 20 gallons per minute to the top of the tower (in which the furnace gas first enters) at 15 g.p.m. to the second tower and at 5 g.p.m. to the third. These quantities are approximate but represent the full amount of magnesium hydroxide slurry going to the towers from both recovered and added magnesium oxide. The absorbing liquor is circulated at about 1000 g.p.m. from the bottom of each tower back to the top of that same tower by vertical type pumps operated at 50-hp., 1200-r.p.m. synchro-



Top level view of heat and chemical recovery furnace

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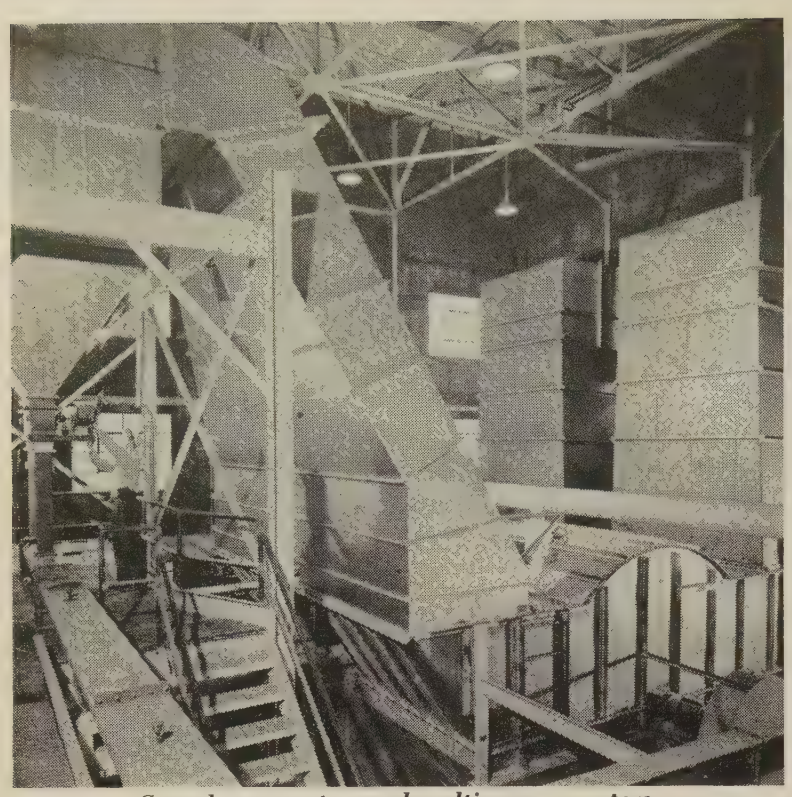

Cascade evaporators and multicone separators
Operator is adjusting gate valve through which recovered MgO passes into screw conveyer.

nous motors. Most of the make-up water is added to the last tower.

Theoretically, a sufficient quantity of sulphur dioxide would be available from the burning of the strong red liquor to react with the recovered and added magnesium oxide to form magnesium bisulphite with little or none of the monosulphite salt. Actually, the magnesium sulphite is in the range 0 to 0.3% and is controlled by regulating the quantity of make-up sulphur dioxide introduced into these towers. Under the existing conditions (pH of 4 to 5) calcium salts are in their most insoluble state and the calcium picked up from original wood and make-up magnesia is removed by a sand filter.

The raw acid is then fortified in two absorption towers with sulphur dioxide produced from sulphur in a small rotary burner. One tower is constructed of concrete with tile lining and operated at atmospheric pressure; the second, which is operated at a pressure of 15 p.s.i., is of steel construction, lined with acid-resisting tile, and packed with ceramic partition tile.

Materials of Construction

The corrosive nature of the acid cooking liquor requires stainless steel in all equipment where contact with metal occurs. Type 316 stainless steel is used in all tubing and pipes that convey the liquor throughout the cooking and recovery plants. Type 316 is also used in all pumps and processing equipment where metal contact is made. Acid-resisting brick and tile are used as linings for digesters, dump tanks, and absorption and acid-fortifying towers, which are constructed of steel plate and/or concrete. Litharge and glycerol or an acid resin jointing is used in cementing of linings. Wood stave tanks are used at the points indicated.

Over-all Operation and Recovery

In the operation of the Longview mill to the present time, chemical consumption has been abnormally high and steam generation below that expected owing to the lack of sufficient evaporator capacity to evaporate all the red liquor available. The mill is currently producing 300 to 325 tons of unbleached pulp per day, whereas the recovery system was designed for a 275-ton-per-day output.

Most representative figures for operations over appreciable periods of time show the magnesium oxide consumption to be 50 pounds per ton of pulp produced. Sulphur consumption, also higher than was anticipated, is 85 pounds per ton of pulp.

With sufficient evaporator capacity, which is presently being installed, the magnesium oxide consumption is expected to drop to 20 pounds and sulphur consumption to 50 pounds; steam generation is expected to increase to 9000 pounds of steam at 615 p.s.i. and 725°F. per ton of pulp. A future report will describe recovery after these changes are complete and operating data have been accumulated. Calcium-base pulping requires 270 pounds of sulphur and 350 pounds of lime rock per ton.

Magnesium oxide pulping, though not yet developed to a state of perfection, is definitely a solution to the sulphite pulp making stream pollution problem. The process is one which fully utilizes the energy content of the organic waste and recovers the chemicals used in cooking; all products recovered are immediately used by the pulp industry. Until such time as markets develop for products that can be recovered from waste liquor solids by other processes and that are economically of greater value than their use as a fuel, the magnesium oxide process offers sound utilization of sulphite waste liquors and elimination of stream pollution. The increased evaporator capacity now being installed at Longview, Wash., is expected to result in greater recovery of cooking chemicals and will further enhance the economics of the process.

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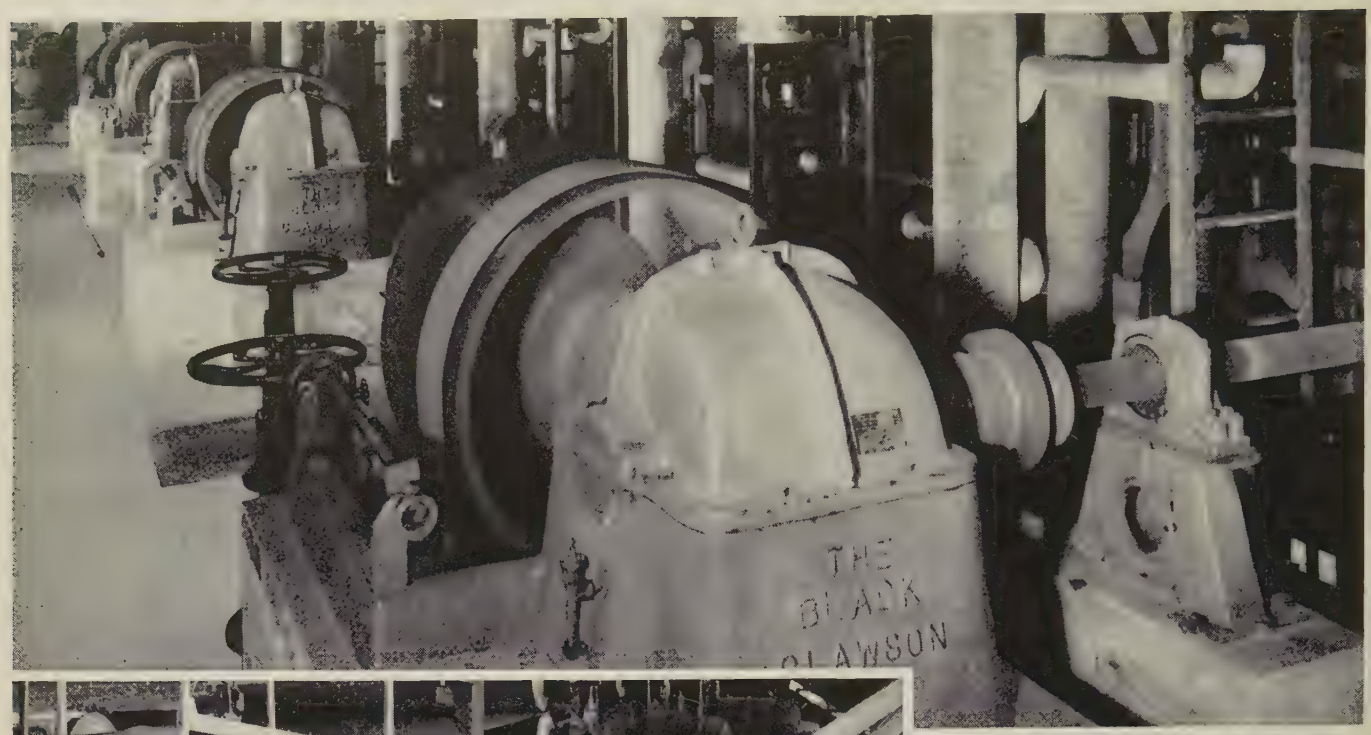

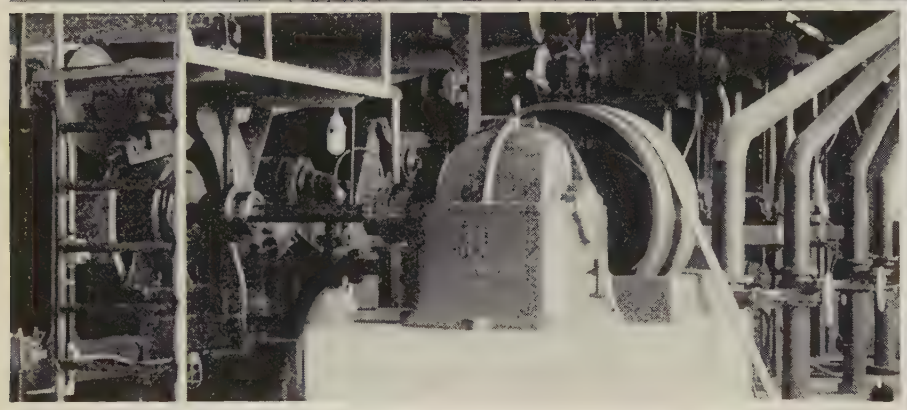

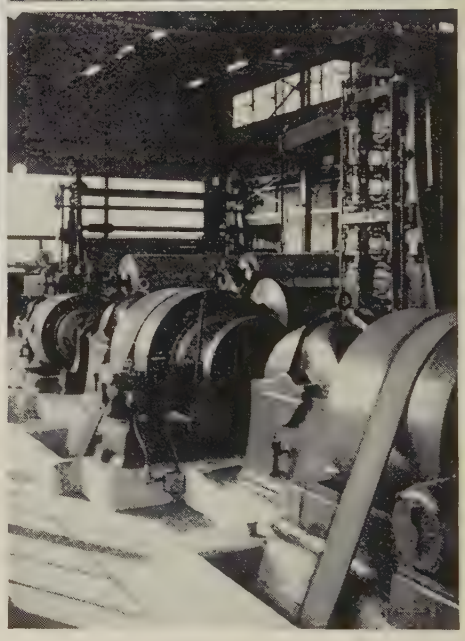

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Ash and Flue Gases from Burning Sulphite Waste Liquor

AXEL SCHOLANDER

Amounts and concentrations of ash and sulphur dioxide from sulphite liquor fuel are reported and discussed. The composition of the ash is given and discussed with respect to the burning of the liquor and the troubles observed in the boilers due to deposits in the superheater. A new furnace and modifications in the fire room have led to good burning conditions. The damaging effects of sulphur dioxide are discussed. Possibilities of elimination of sulphur dioxide by a scrubbing process have been studied on a laboratory scale and measured in some industrial scrubbers.

THE problem of evaporating and burning sulphite waste liquor in a technical and economical way can be said to have been solved during the last decennium. Primarily it is the incrustation problem which has been successfully attacked by engineers and scientists but many problems are still waiting for a better solution on the reclaiming side and on the burning product side.

In Sweden, at present, 14 mills have installed evaporation stations. The total capacity will be 283 tons of 10% waste liquor per hour which corresponds to about 15% of the total liquor output in Sweden.

Of these 14 mills nine evaporate neutralized liquor (three and a half liquor from strong pulp, five and a half liquor from silk pulp) and five acid liquor. Three mills in the first group and one in the second evaporate fermented liquor and combine evaporation and stripping of the alcohol in the liquor.

The biggest plant yet installed was started in December, 1950, at Mo & Domsjö AB, Örnsköldsvik. It is built by AB Rosenblads Patenter, Stockholm, and has a capacity of 40 tons of water evaporated per hour. It runs as a pure counterpressure station, and the low-pressure steam obtained is consumed in the alcohol plant and in the digester department. The liquor is burned in the boiler department either together with coal powder or alone in a special furnace mentioned below. An amount of 16,000 tons of coal will be saved per year.

However, attention has been directed to the unpleasant secondary phenomena connected with the ash and the flue gases from the burning of sulphite waste liquor.

ASH AND SULPHUR DIOXIDE FROM SULPHITE LIQUOR FUEL

Evaporated sulphite liquor is an extraordinary fuel which is perhaps best illustrated by a comparison with various other fuels on a calorie basis.

Most striking is the large amount of sulphur dioxide which, by burning sulphite liquor, can reach values six to eight times those obtained by burning oil or coal. The ash amounts will be about twice those obtained when coal is fired—all amounts calculated on the assumption of production of the same amount of heat.

AXEL SCHOLANDER, Mo & Domsjö AB, Örnsköldsvik, Sweden; Sulphite Waste Liquor Evaporation Committee, Cellulosaindustriens Centrallaboratorium, Stockholm, Sweden.

COMPOSITION OF SULPHITE LIQUOR FUEL

Sulphite liquor fuel is usually produced in one of two forms—50 to 55% thick liquor or dry powder. Calculated as a percentage of the dry substance both forms will contain:

Total CaO	5–8%
Total S	5–8%
Ash content	7–15%

The values vary with pulp quality, neutralization, and evaporation conditions. The lower limits correspond to waste liquor, not neutralized before evaporation: the higher limits to neutralized liquor from the alcohol plant where lime or lime mud have been added in order to adjust pH before fermentation.

The amount of dry substance in the liquor per ton of pulp will, of course, vary greatly with the pulp quality. For the calculations below, it is assumed that 1 ton of dry substance in the liquor is obtained per ton of dry pulp.

Fly Ash Content in Flue Gases. As mentioned before, sulphite liquor fuel can be burned as thick liquor or dry powder. In both cases, the ash will appear as fly ash and follow the flue gases. A normal composition of the combustible substance is:

C	50.8%
H	5.2%
S	5.6%
O	38.4%
	100.0%

With an ash content of 10% and carbon dioxide concentration of 15% (75% of theoretical concentration) a fly ash amount of 18 grams per Nm^3 in dry gases is observed when sulphite liquor fuel is burned alone. If mixed fuels are used, lower values are obtained.

Fly Ash. The ash content in the dry substance was 7 to 15%, which corresponds to 70 to 150 kg. fly ash per ton of pulp.

COMPOSITION OF THE ASH

The composition of the ash from sulphite liquor

Table I. Ash and Sulphur Dioxide from Various Fuels

Amounts Calculated per 1000 Cal.

Fuel	*Ash, grams*	*SO_2, grams*
Coal Ash, 15% Sulphur, 1% Heat value, 6,000 M cal./ton	25	3.3[a]
Oil Ash Sulphur, 2% Heat value, 10,000 M cal./ton	..	4.0[a]
Sulphite liquor fuel (50%) Ash, 8% Sulphur, 3% Heat value, 1,850 M cal./ton	43	24[b]

[a] All of S forms SO_2.
[b] 75% of S forms SO_2.

Table II. Ash Compositions

	Sulphite liquor only (50%)	*Sulphite liquor only (50%) (Loddby furnace)*	*40% Sulphite liquor: 54% wood*
$Fe_2O_3 + Al_2O_3$	7	1	3
$CaSO_4$	36	54	46
$CaCO_3$	42	3	28
CaO	14	38	22
CaS	1	1	1
$CaSO_3$	..	2	..

fuel, burnt in full-scale installations, has not been reported in the literature.

The main components of the ash are $CaSO_4$ (anhydrite form), CaO, $CaCO_3$, CaS, and $CaSO_3$. Besides, small amounts of sesquioxides and silica are usually present, and, depending upon the firing conditions, unburnt in the form of coal.

The proportions between the main components vary somewhat, and the examples given below clearly illustrate this point.

The composition of the ashes is interesting. The presence of $CaSO_3$ is somewhat astonishing, as the reaction:

$$4CaSO_3 = CaS + 3CaSO_4$$

sets in at 650°C. Over 900°C. $CaSO_3$ dissociates

$$CaSO_3 = CaO + SO_2$$

This dissociation is perceptible at 650°C. and almost complete at 1100°C. The behavior of $CaSO_3$ at different temperatures has been studied by Foerster and Kubel (*1*).

The composition of the ash and its properties will be discussed later on in this article.

It may be pointed out that determination of coal in the ash cannot be made as ignition loss as the dissociation of $CaCO_3$ will then cause erroneous results. Correct results are obtained by the following method:

About 1 gram of ash is weighed out and boiled 5 minutes with 100 ml. of 1 *M* HCl. Filter and boil again 5 minutes with 100 ml. of 1 *M* HCl. The residue is filtered off on a glass filter, washed with water, and dried at 180°C. and weighed. Part of the dry residue is weighed and ignited in a porcelain crucible. The residue is moistened with some drops of conc. sulphuric acid in order to transform CaS to $CaSO_4$, again ignited, and weighed.

ASH DEPOSITS IN BOILERS

The risk of ash deposits in the boiler has been discussed by Brunes (*2*). Since this article appeared, a new furnace for burning 50% sulphite liquor has been constructed at Holmens Bruks AB in Loddby, Sweden, and patents applied for by AB Rosenblads Patenter. This furnace resembles a cyclone burner for coal powder fuel.

The cylindrical furnace (length~2 m., diam.~1.5 m.) is not cooled. The air is introduced tangentially at great velocity; the fuel is fed by a single spray nozzle (Nagelburner with steam atomization) mounted centric and axially. The combination of the radiation and the great turbulence from the uncooled walls causes a rapid drying of the droplets and a good combustion.

In fact, as low values as 0.5 to 1.0% of coal in the ash are observed. Burning in usual boiler furnaces gives between 5 to 15% coal in the ash. These values all refer to the fly ash. The ash, falling in the bottom of the furnace, usually contains no coal as its retention time is very long, thereby securing complete combustion. Samples of this ash are therefore not representative for the over-all combustion result.

According to Brunes, risk for ash deposits in the superheater and economizer is diminished when 50% liquor is burnt instead of dry powder. Deposits have, however, been observed even in boilers fired with 50% liquor. The deposits have a brittle, powdery character and are easy to remove. Their structure is sometimes laminar showing that layers of sticky ash have been covered with non-sticky ashpowder. The factors causing stickiness have not been ascertained as yet. Zinsen (*3*) has studied the melting properties of different ashes and included brown coal ashes, the character of which very much resembles ash from sulphite liquor, burned for the calcium and sulphur content. He has shown that a eutecticum exists between CaS and $CaSO_4$ at 830°C. This might serve as an explanation of the stickiness. During the combustion process the sulphur content of the liquor is partly reduced and later on oxidized to sulphite and sulphate. Melted ashdrops of sulphide-sulphate may exist and cause the stickiness.

If iron is present this will cause a higher melting point of the ash through the reaction:

$$2CaSO_4 + Fe_2O_3 \rightarrow Ca_2Fe_2O_5 + 2SO_3$$

whereby the melting point will rise to 1200°. The alkali salts in the wood, especially from salt water floated timber, might also be responsible for the stickiness.

If coal is burnt together with sulphite liquor fuel less deposits are observed. Whether this fact is due to the reaction above or to the grinding and mechanical effects of the coal powder fly ash has, however, not been stated.

The difficulties due to deposits in the superheater and the first convection parts in the boiler at Domsjö, which is equipped with Loddby furnaces, have now been largely overcome since Mr. Sandberg, the power engineer, has made changes in the boiler. New cooling surfaces are installed in the form of La Mont tubes, arranged as screens in the upper part of the fire room, parallel to the gas stream. The side walls and the bottom are completely covered with La Mont tubes. The sticky ash particles give off radiant heat to the tubes and screens and are thereby cooled below the danger temperature limit for stickiness.

The boiler now runs continuously on sulphite liquor fuel (50 to 55%) without any supplementary fuel and gives an amount of 14 tons per hour of steam.

SULPHUR DIOXIDE

When burning sulphite liquor fuel of the composition reported above, the theoretical CO_2 content of dry gases will be 19.8% and the SO_2 content 0.82%.

In practice, values of 0.5% SO_2 at CO_2 = 13 to 15% have been observed.

When mixed fuels are burned, the sulphur dioxide content in the gases will depend upon the supplementary fuel and its sulphur content.

When burning sulphite liquor fuel, it will be shown later on that 70 to 80% of the sulphur will appear as sulphur dioxide in the flue gases, and 30 to 20% re-

main in the ash, mainly as sulphate. According to the conditions assumed above, 70 to 130 kg. of SO_2 are produced per ton of pulp.

The sulphur dioxide produced may cause damage to the boiler as well as the environment of the factory.

The troubles likely to be encountered in the boiler do not depend as much on SO_2 as on the small amounts of SO_3 in the gases. This problem has been treated thoroughly by Johnstone (*4*), Liander (*5*), and, in the case of sulphite liquor burning especially, by Brunes (*2*) and Rosenblad (*6*). In these articles it is shown that a raised dew point is observed, however, not catastrophically high. Values 20 to 30° over the water dew point have been observed. No definite corrosion effects for which the sulphite liquor fuel could be solely responsible have been observed. The strongly alkaline fly ash will, of course, immediately neutralize any acid condensates on the tubes.

The damage caused by sulphur dioxide in industrial areas has been intensely investigated (*7*, *8*). In this case, the sulphur dioxide sources were of a capacity 30 to 40 times greater than that of a sulphite mill producing 40,000 tons per year.

The investigations show that damage to vegetation is strongly dependent on the season of the year, topographical and meteorological conditions. Low but constant sulphur dioxide concentrations (<0.2 p.p.m.) appear to be not as dangerous as exposures of shorter durability but higher concentrations. The risk for damage is greatest in the vegetation period when the physiological activity has a maximum. High relative humidity, low temperature, and consistent wind velocity from a constant direction will favor damage.

In order to judge the risk of damage caused by sulphur dioxide, a simple apparatus according to Liesegang (*9*) has been used. A Soxhlet-extraction cylinder is moistened with an alkaline solution and placed at strategic points around the factory. After 1 week's exposure the cylinders are analyzed and the sulphur absorbed is determined and taken as a measure of the risk of damage.

Liesegang states that, according to his experience in areas where sulphur dioxide in amounts up to 70 to 100 mg. per week are indicated, there is no risk of damage to the vegetation. At Mo & Domsjö observations have been carried out a year before the burning of sulphite liquor started. The absolute maximum value observed was during this period 20 mg. SO_2 per week maximum. For most weeks values of 3 to 10 mg. SO_2 per week are obtained. After the evaporation and burning started, the absolute maximum value observed has been 38 mg. SO_2 per week. Both maximum values were measured at a distance 1 km. from the chimney.

It would, of course, be better to measure the sulphur dioxide concentration in the air directly by means of some suitable instrument. These are, however, expensive and the value of single determinations in time and place is doubtful. The relative value obtained with the Liesegang clocks is an average value integrated over longer time periods. The absorption process is about the same as when sulphur dioxide is taken up by vegetation in leaves and needles.

SCRUBBING OF FLUE GASES FROM SULPHITE LIQUOR BURNING

The large amounts of sulphur dioxide and ash will actualize a recovery system. Investigations have, however, shown that the sulphur dioxide recovery is not economical as the gas is too dilute. The work on this subject has therefore been concentrated on elimination processes.

A scrubbing process, where the acid gases and the alkaline ash neutralize each other, has been proposed and studied.

The CaO, $CaCO_3$, and CaS in the ash are thereby used for absorption of CO_2.

$$\begin{aligned} CaO + 2CO_2 + H_2O &= Ca^{2+} + 2HCO_3^- \\ CaCO_3 + CO_2 + H_2O &= Ca^{2+} + 2HCO_3^- \\ CaS + 2CO_2 + 2H_2O &= Ca^{2+} + 2HCO_3^- + H_2S \end{aligned}$$

and then of SO_2

$$\begin{aligned} SO_2 + HCO_3^- &= HSO_3^- + CO_2 \\ SO_2 + 2HCO_3^- &= SO_3^{2-} + H_2O + CO_2 \end{aligned}$$

The SO_2 is partly oxidized to SO_4^{2-} in the solution

$$SO_2 + {}^1/_2O_2 + H_2O = 2H^+ + SO_4^{2-}$$

No more SO_2 is absorbed when all CaO, $CaCO_3$, and CaS is transformed into $Ca(HSO_3)_2$ (without oxidation) or $CaSO_4$ (with oxidation). It is easy to see that twice as much sulphur dioxide can be absorbed without oxidation as with oxidation. Therefore the oxidation conditions are important. Oxidation is favorized by high temperatures and presence of certain catalysts (Fe, Mn, etc., ions) (*10*, *11*). This catalyzed oxidation is, however, not so important in practice as long as inhibitors are present both in the gases and in the solution.

Assuming that no catalyzed oxidation takes place, the main question is: what amount of sulphur dioxide from a given amount of sulphite liquor can be absorbed by its own ash? This question can be answered when the partition of the sulphur between gas and ash is known.

Analysis of a liquor and its ash gave the following results:

Sample	CaO_{total}	S_{total}	*mol. S/mol. Ca*
Liquor	2.96	2.78	1.64
Ash	50.2	10.2	0.36
Gas (calc.)	...	...	1.28

Thus, in the ash, 36% of the calcium is combined with sulphur as sulphate (or sulphide) and 64% as carbonate or oxide. The absorption capacity of the ash will be 1.00 −0.36 = 0.64 eqv. per mol., which corresponds to absorption of, without oxidation (abs. to $Ca(HSO_3)_2$):

$$2 \times 0.64 \text{ mol. S/mol. Ca} = 100\% \text{ of } SO_2 \text{ in the gas}$$

with oxidation (abs. to $CaSO_4$):

$$0.64 \text{ mol. S/mol. Ca} = 50\% \text{ of } SO_2 \text{ in the gas}$$

The absorption capacity of the ash can be determined separately. Excess hydrochloric acid is added, the solution is boiled, and the acid is titrated with alkali and bromcresol green-methyl red indicator.

SCRUBBING OF FLUE GASES IN PRACTICE

In practice, these theoretical limits can of course not be reached. Such a flue gas scrubber, combined with wet separation of the ash, may be looked upon as an

acid tower working under very bad circumstances: the gas is hot and of low quality, and the contact gas-liquid is not good. The consequence is that the efficiency is low, and the oxidation of absorbed sulphur dioxide is high.

Studies of the working conditions of a flue gas scrubber showed an absorption of 30% of SO_2: 60% of the absorbed SO_2 was oxidized in the solution. However, only 50% of the absorption capacity of the ash was used which shows that the efficiency of the scrubber can be increased by installing more absorption surfaces.

The scrubber was a rectangular wooden tower with sections of wood grids.

The main scrubber data were

Gas	=	4400 $Nm^3/m^2.h$
Water	=	1.5 $m^3/m^2.h$
Circulating liquor	=	1.5 $m^3/m^2.h$
Surface of grids	=	12 m^2/m^2

The scrubber was installed in order to serve as wet dust separator and not primarily as an absorption unit. The dust separation efficiency was quite good. The ash water obtained contained 1 to 2% solids and had a temperature of 50 to 55 °C. It passed a small settling tank (in order to prevent clogging of the heat exchanger) and a heat exchanger before it was pumped out.

Recent experiments show that better absorption can be obtained using other types of absorption equipment.

In many cases sulphite waste liquor evaporation and burning are planned in order to avoid water pollution by the waste liquor.

Burning of the liquor might transform the problem to an air pollution problem. As has been shown above, the risk of sulphur dioxide damage is relatively small. The ash might cause troubles in the neighborhood of the mill—as does also the fly ash from coal powder firing. The effects of sulphite liquor ash on vegetation will, in most cases, be beneficial as it will act as a lime and sulphur fertilizer. This might be one of the potential uses of the ash.

If a scrubber process is used, the problem is again transformed to a water pollution problem. This time, however, the effluent contains no oxygen-consuming substances. The effect on the recipient will be that its concentration of calcium and sulphate will rise which is harmless if anaerobic conditions caused by other sources are not prevalent.

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The Statistical Calculation of Bursting Strength

E. R. HOFFMAN

With the advent of statistical quality control to a paper mill, the preparation and maintenance of many control charts for the properties of the grades manufactured become a problem. One chart may be used for each property when data are transposed to hypothetical values based on a standard grade of paper. The method is illustrated by Mullen data and consist of graphically establishing the relation of bursting strength to the weight of the paper and the various constituents of the furnish. The data are then adjusted mathematically to values which would have been obtained if the paper machine had been manufacturing one grade and weight of paper. The resultant chart may then be used for the study of paper machine performance, beater standardized beating procedure.

In the application of statistical quality control to a paper mill one runs into the difficult requirement of many charts for the qualities of the grades manufactured. It is desirable, therefore, to use one chart showing the complete story of a particular property. The problem then becomes that of reducing all data related to a property to a common denominator. In order to do this it is necessary to determine with mathematical precision the effect of various factors of which the property is a resultant. This paper deals with such a method and embraces the reduction of bursting strength measurements taken to a common hypothetical value based on a standard furnish, also the plotting of these data as a control chart and the use of the data in the design of furnishes or tolerances.

The bursting strength data used here cover a 30-day period, taken at machine conditions, and are plotted in Fig. 1. The erratic variation in values readily demonstrates the effect on burst of factors such as weight and furnish. An analysis of the data shows that weight is the biggest single cause of variation of the strength values. In order to determine the relation between weight and bursting strength, the average values for the last four years, for various weights and grades, have been plotted against the nominal weights, based on a 17 by 22-inch sheet. These grades include bond, ledger, mimeo, cover, offset, etc., and fall into four categories as shown in Fig. 2. When plotted on semilog, the curves become linear, Fig. 2A.

The exponential curves of Fig. 2 can be explained

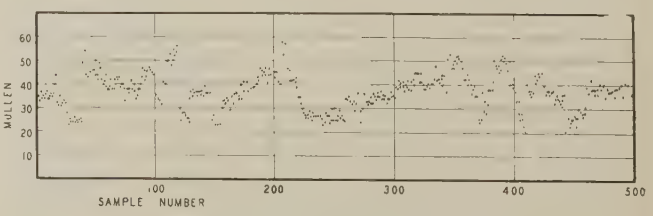

Fig. 1. Bursting strength versus sample number (time), no corrections

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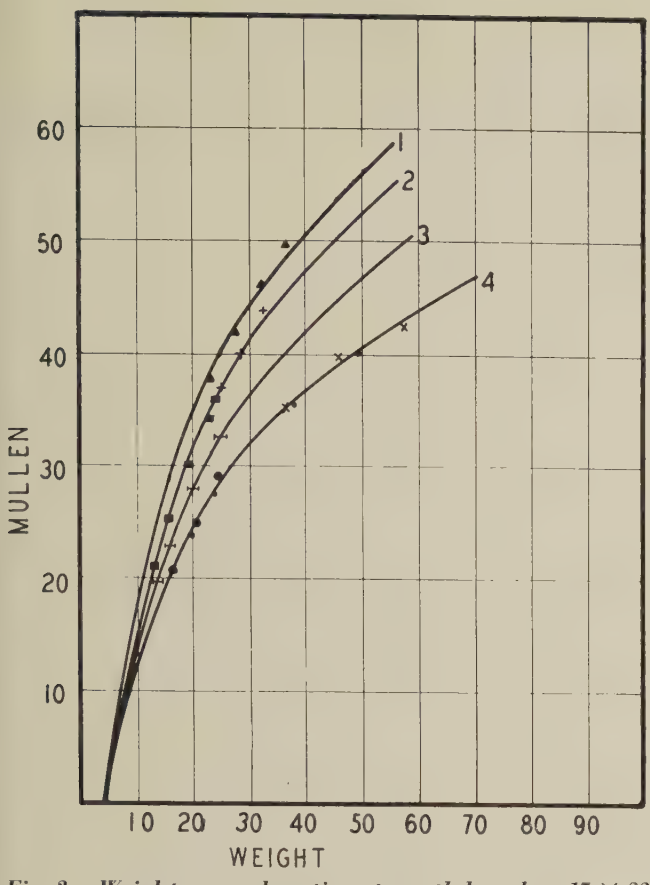

Fig. 2. Weight versus bursting strength based on 17 × 22 ream size

as follows: starch from surface sizing, as will be shown later, increases bursting strength. The depth and quantity of starch on a sheet of paper are the same for normal sizing conditions regardless of the thickness of paper being treated. It therefore follows that the starch-fiber ratio of a thick sheet is lower than for a thin sheet. The heavy sheet, primarily all fiber, therefore, has a lower burst value per unit weight

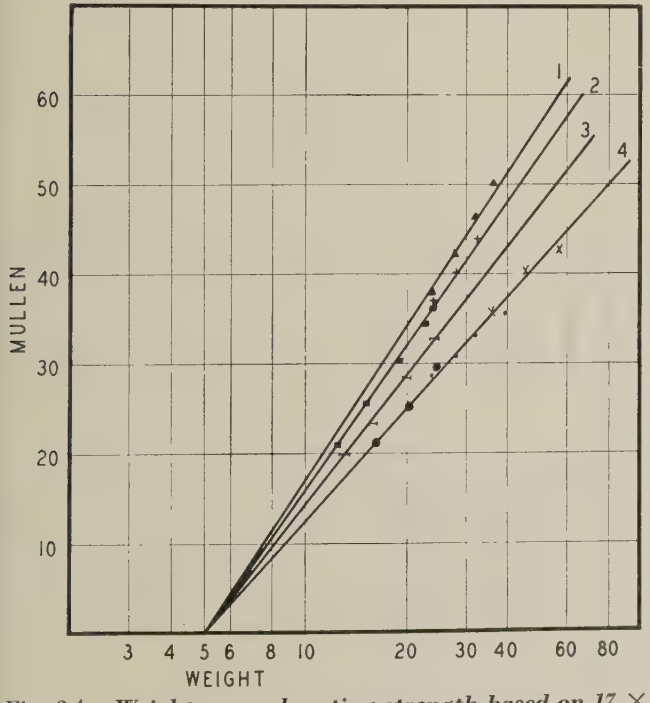

Fig. 2A. Weight versus bursting strength based on 17 × 22 ream size

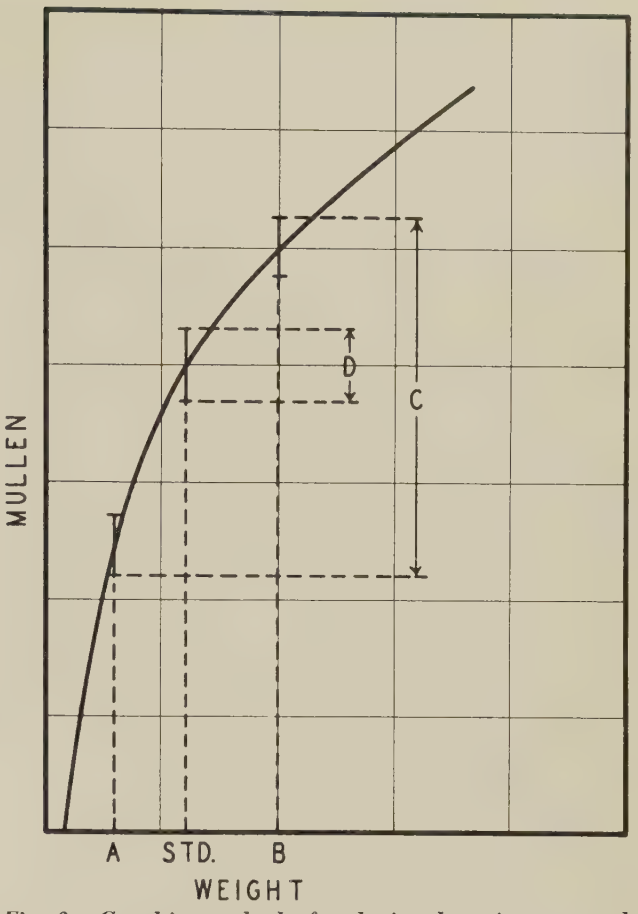

Fig. 3. Graphic method of reducing bursting strength values to standard hypothetical values

than the thin sheet which contains a higher ratio of starch and a correspondingly higher burst value per unit weight. Thus, the exponential bursting strength-weight curve reflects the changing ratio of starch to fiber for papers of various weights.

Further study of the data show that the previously mentioned grouping of papers in the weight-burst relation was the effect of varying amounts of filler and size in the papers. Papers with lower filler content developed a higher burst-weight relation than highly filled sheets.

From the relations established for various papers in Fig. 2, the strength measurements presented in Fig. 1 were reduced to values comparable to a common weight of 24 pounds based on a 17 by 22-inch sheet. The actual operation was simply performed by adding to or subtracting from the measured burst, as the case required, the difference in burst values for the paper being considered and the standard weight as determined from the curves. Figure 3 illustrates graphically the method. It shows how case A and B with a range C is reduced to range D. In Fig. 4 the paper machine

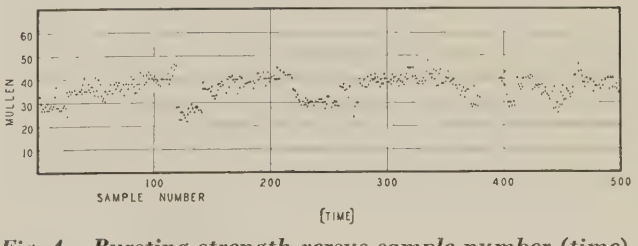

Fig. 4. Bursting strength versus sample number (time), corrected to 24-pound weight; 17 × 22 ream size

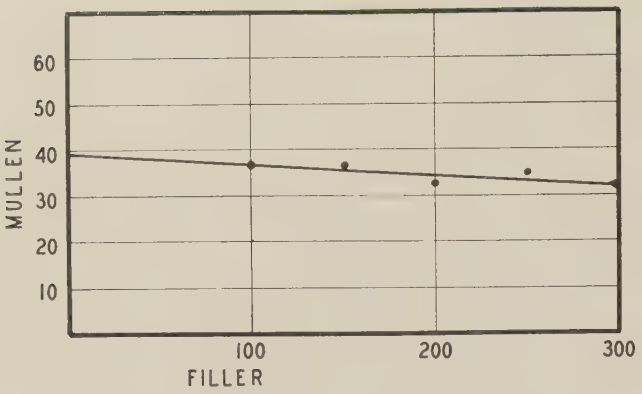

Fig. 5. Bursting strength versus filler

data previously shown in Fig. 1 have been thus treated. Substantial reduction in the erratic variation has been accomplished. It is evident from the remaining variation, however, that other variables still exist. Standardization factors for filler, surface sizing concentration, and additions of various types of pulps have also been determined (see Figs. 5 and 6). These were obtained by plotting the bursting strengths, after adjusting the values to a common weight and ream size, against the amounts of the various constituents in the

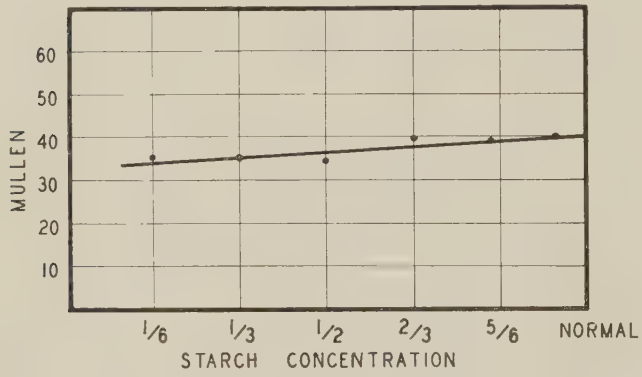

Fig. 6. Bursting strength versus surface size concentration

furnish. As the relation for filler was determined all data were then readjusted to a standard furnish of no filler and then the data replotted before proceeding with the next variable. In Fig. 7 standardizations for weight, ream size, and filler have been made. Figure 8, after adjustment for starch content, then represents the values that would have been attained if the paper machine had been making only a 24-pound sheet using an all sulphite spruce fiber, no filler, and a normal starch concentration.

It was found that variations in rosin, alum, and the rosin-alum ratio had practically no effect on bursting strength, but that certain types of fibers did have an appreciable effect and had to be taken into account.

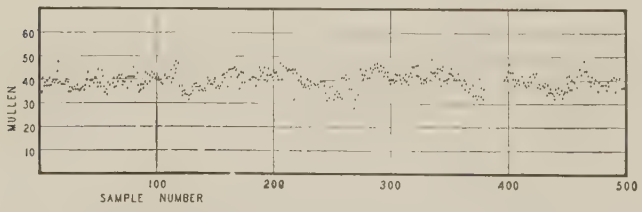

Fig. 7. Bursting strength versus sample number (time), corrected to 24-pound weight; 17 × 22 ream size; no filler

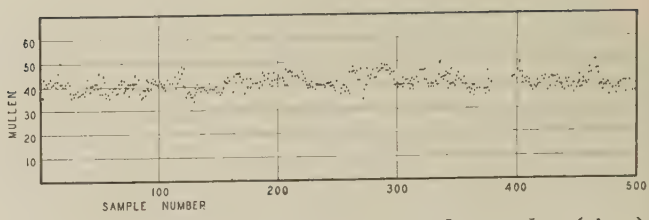

Fig. 8 Bursting strength versus sample number (time), corrected to 24-pound weight; 17 × 22 ream size; no filler; normal surface sizing

The accumulated, standardized data, when plotted on an appropriate control chart shows a remaining variation (see Fig. 9). The magnitude of the remaining variation is of sufficient size so as to occasionally indicate other assignable causes of variation or out of control. These appear small during the period studied and of minor importance. Seasonal changes, however, may be larger and of considerable interest. A number of variables have not as yet been studied to determine whether or not they have an appreciable effect on bursting strength variations. For example, in this group may be calendering, moisture of the paper at various points throughout the paper machine, felting as the result of shake, etc. Although beating is known to have a profound effect on burst, this study indicates

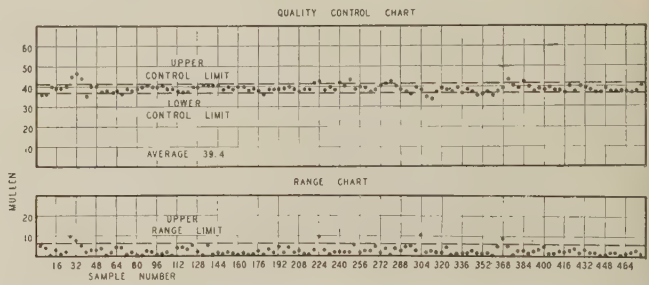

Fig. 9. Quality control and range chart for bursting strength

that beating during the routine manufacturing period was reasonably uniform. The average level of the burst value is probably to a great extent a measure of the degree of beating. If the beating cycle is changed or beating procedure altered one could probably detect the effectiveness of such changes.

The common control chart using the hypothetical values is important because it makes possible the comparison of the performance of a paper machine from time to time regardless of the paper being manufactured, or the comparison of two or more machines also regardless of grade. It opens the way to a means of checking beater operation with respect to bursting strength, or if a standardized beating procedure is used one could study the reaction of various pulps to beating in the mill process.

Once the average hypothetical burst value has been established for the process, the calculation may be reversed and the expected burst value for any furnish or weight may be determined. With a knowledge of the normal process deviation, the appropriate range about the expected value may be established. These limits would be the appropriate tolerance for a given furnish.

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Effect of Bark on Yield and Quality of Sulphate Pulp from Southern Pine

J. S. MARTIN and K. J. BROWN

Sulphate pulping tests were made on shortleaf pine chips without bark, on bark alone, and on mixtures of chips and bark containing up to 24% of bark by weight. These tests indicate that considerable percentages of bark can be used in kraft-type pulps but that sulphate pulps made from bark only are unsuitable for use in paper products. In the production of kraft-type, bleaching sulphate, and sulphate semichemical pulps, the presence of bark caused (1) an increase in yield of pulp from a cord of rough pulpwood, (2) an increase in consumption of active chemicals during cooking, and (3) a decrease in brightness of the pulp, or darker-colored pulps.

THE possibilities of reducing barking costs, improving wood-storage practices, and obtaining larger yields of pulp from the same amount of pulpwood by using part or all of the bark have revived interest in the effect of bark in pulping. Before pine is pulped by the sulphate process, it is common practice in paper mills to pass the pulpwood through barking drums to remove most of the bark from the wood. Some mills remove all of the bark, while others allow sizable amounts to remain on the pulpwood. The bark is removed because it has been considered detrimental to pulping, though very little specific information is available on how much bark is undesirable.

A systematic investigation was made to determine the effect of bark on the quality and yield of pulp. Pulping tests by the sulphate process were conducted on shortleaf pine and its bark over a range of wood-to-bark ratios. The main emphasis was placed on the production of kraft-type pulps, such as those used for wrapping paper and paper bags. Bleaching sulphate and sulphate semichemical pulps were also made from mixtures of wood and bark.

A difference in the pulping characteristics of wood and bark would be expected because of the difference in their chemical natures. The cellulose in the bark fiber appears to be combined with lignin by a linkage that is very resistant to splitting by either caustic soda or phenol and acid (*1*). This linkage is claimed to be stronger than the ligno-cellulose bond in wood, which is considered to be only a physical union. The tenacity with which lignin is held to the cellulose in the bark indicates an actual chemical union. Although caustic soda does not readily split this chemical union, it quickly dissolves bark tissue. The high solubility of bark in 1% sodium hydroxide and in hot water are indications that low pulp yields can be expected from bark.

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TEST PROCEDURES

Description and Preparation of Material

Unpeeled shortleaf pine pulpwood of 6 inch average diameter was received at the U. S. Forest Products Laboratory from Crossett, Ark. The bark was separated from the wood as close to the cambium layer as possible.

Microscope slides of macerated tissue from bark sections were prepared by allowing the bark to stand for 6 hours in a mixture of 10% nitric and 10% chromic acids. The bark tissue was then washed, placed on a slide, and stained. The cell structure of the macerated bark tissue was examined with a microscope under fluorescent light. Figure 1 is a sketch of the material as seen through the microscope, and Fig. 2 shows the structure of the cells present in the bark tissue.

The peeled wood was reduced to $^5/_8$-inch chips by the Laboratory chipper. The bark strips were cut into chips of the same size with a paper cutter. Wood and bark chips were mixed together in proper proportion before each digestion.

Data on the physical and chemical properties of the wood and bark are given in Table I. The chemical tests were made by methods used primarily for wood analysis.

Cooking Conditions for Sulphate Pulping

The digestions were made in steam-jacketed, spherical, rotary autoclaves of 0.5 cubic foot capacity. Three series of duplicate digestions were made using charges that contained 100 and 0, 92 and 8, 84 and 16, 76 and 24, and 0 and 100% of wood and bark, respectively, based on the total weight of moisture-free bark and wood. Each series of digestions was made with different amounts of cooking chemicals to give semichemical, kraft, and bleaching types of pulps. The semichemical pulps in the 60% yield range, based on the weight of the wood, were fiberized in an attrition mill having a single rotating disk of 8 inch diameter. Constant cooking conditions for all digestions were: liquor-to-wood ratio, including moisture in the chips, 4 to 1; temperature-pressure increase period from 30°C. to maximum temperature of 170°C., 1.5 hours; duration at maximum temperature, 1.5 hours; and sulphidity, based on active alkali, 30%. The other cooking conditions are given in Table II. The physical and chemical properties of all the pulps were tested by standard methods, and the data are given in Table III.

DISCUSSION

The Bark

Shortleaf pine bark has one-half the cellulose content

and nearly twice the lignin content of shortleaf pine wood and is three times as soluble in hot water and nearly four times as soluble in 1% sodium hydroxide according to the test methods used (Table I). These relationships, especially, the high solubility in alkali, indicate that low pulp yields and high chemical consumptions will be obtained when pulping bark.

Table I. Physical and Chemical Properties of Shortleaf Pine Wood and Bark[a]

Property	Material: Wood	Material: Bark
Physical properties[b]		
Specific gravity[c]	0.493	...
Density,[c] (lb. per cu. ft.)	30.8	...
Diameter of log, in.	6.0	...
Age of log, yr.	27.1	...
Rate of growth, rings per inch	9.2	...
Summerwood, by volume, %	36.5	...
Compression wood, by volume, %	3.0	...
Heartwood, by volume, %	4.6	...
Bark, by volume, %	...	11.0
Bark, by weight, green basis, %	...	7.0
Bark, by weight, moisture-free basis, %	...	7.7
Chemical composition[d]		
Lignin, %	27.6	45.6
Holocellulose, %	69.3	34.2
Alpha-cellulose, %	48.5	21.9
Total pentosans, %	8.9	7.6
Ash, %	0.5	1.6
Alcohol-benzene extractables, %	3.3	7.2
Ethyl ether extractables, %	2.2	4.6
Hot water extractables, %	2.8	8.5
Sodium hydroxide[e] extractables, %	12.2	44.7

[a] *Pinus echinata.* Shipment No. 2984.
[b] Average of values obtained on transverse sections.
[c] Based on weight of moisture-free wood in a given volume of green wood.
[d] Based on analyses of samples of wood and bark chips.
[e] Water solution of 1% concentration by weight.

The nature of the cellular material forming the structure of bark is a major factor in determining its value as a pulp. An examination of the bark under a microscope showed three types of cells that might give usable fibers (Figs. 1 and 2). The estimated amounts of these materials were 85% of sieve cells, 10% of parenchyma cells, and 5% of stone cells.

The sieve cells were found to be roughly 0.75 mm. in length, or about 25% as long as pine wood fibers and 60% as long as many hardwood fibers. The sieve cells found in the active phloem resembled wood fibers and should have good bonding characteristics in pulps.

The parenchyma cells were short, soft, thin-walled, and brick-shaped. The stone cells were short, quite hard, and nearly spherical in shape. Because of their small size, the parenchyma and stone cells may lower quality and strength. A great portion of these small cells may also be lost during pulping and pulp processing, thus lowering pulp yield.

Sulphate pulping tests indicated that bark alone was not pulped adequately by standard cooking conditions for producing a kraft pulp. Bark gave a greater weight of screening rejects than of screened pulp and had a high chemical consumption, which indicated that the cooking chemicals were nearly exhausted at the end of the digestion (Table II). The use of more drastic cooking conditions, similar to those used for producing bleaching sulphate pulps, reduced markedly the screening rejects but only increased the yield of screened pulp from 15 to 18% on a total-weight basis. Under the same conditions, the pulp yield from wood

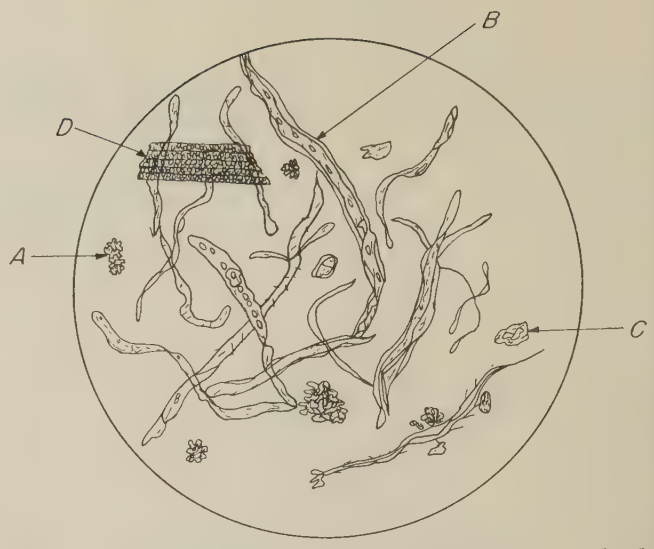

Fig. 1. Sketch of macerated tissue of southern pine bark as observed with microscope

A, stone cells; *B*, sieve tube; *C*, parenchyma (collapsed); *D*, ray section.

alone would be reduced from 48 to 44%. The yield of semichemical pulp obtained from the bark was 42% or almost 30% lower than for wood alone.

Bark sulphate pulps were very dark in color and had a slippery feel. They contained small, hard particles that settled out of water suspensions and caused difficulty in handling, especially in washing and draining on screens. These difficulties were not encountered in pulps made from bark and wood mixtures.

The pulps made from bark alone were much lower in chemical purity than those made from wood as would be expected from the marked difference in the chemical composition of the original materials before pulping. Lignin content was 13% for the bark pulp as compared with 3% for the bleaching wood pulp. The strength of bark pulp was very low. Bursting and tearing strengths were only one tenth of those obtained from pine wood pulp (Table III). The bark sulphate pulps were too low in yield, strength, and freeness for use alone in paper products.

Kraft Pulps

In production of kraft pulps from mixtures of wood and bark, an increase in the amount of bark up to 24%

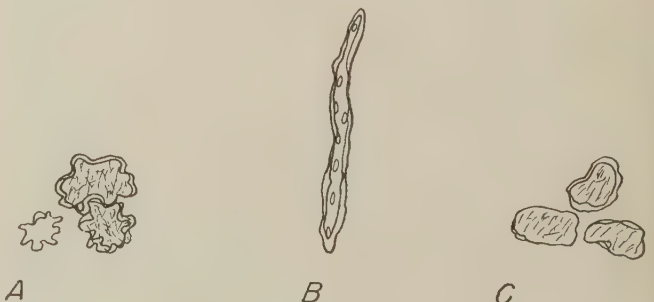

Fig. 2. Structure of cells of southern pine bark as observed with microscope

A, stone cells, which are almost spherical in shape and comprise about 5% of the bark; *B*, sieve cells, which average about 0.75 mm. in length and comprise about 85 to 90% of the bark; *C*, parenchyma cells, which are brick-shaped and comprise about 5 to 10% of the bark.

Table II. Conditions and Results for Sulphate Pulping of Shortleaf Pine Wood, Bark, and Wood-bark Mixtures

Digestion no.	Materials charged, % Wood	Materials charged, % Bark	Cooking conditions[a]: Chemicals charged: Initial concentration calculated as Na_2O, g./l.	Cooking conditions[a]: Chemicals charged: Amount per 100 pounds of moisture-free material, lb. $NaOH$ plus Na_2S	Cooking conditions[a]: Chemicals charged: Amount per 100 pounds of moisture-free material, lb. Calculated as Na_2O	Chemicals consumed, %	Black liquor density at 15°C., °B.	Yield of moisture-free pulp per 100 pounds of moisture-free material, lb. Screened	Yield of moisture-free pulp per 100 pounds of moisture-free material, lb. Screenings	Yield of moisture-free pulp per 100 pounds of moisture-free material, lb. Total	Brightness of pulp, % Unbleached	Brightness of pulp, % Bleached[b]	Permanganate number of pulp
Bleaching Sulphate Pulps													
2241x, 2242x	100	0	53.7	27.50	21.5	67.8	12.7	43.7	0.1	43.8	26.6	68.0	16.0
2243x, 2246x	92	8	53.7	27.50	21.5	71.0	12.9	41.6	0.1	41.7	21.7	64.0	16.5
2247x, 2248x	84	16	53.7	27.50	21.5	73.5	12.9	39.7	0.2	39.9	15.1	57.9	17.9
2249x, 2250x	76	24	53.7	27.50	21.5	75.9	13.3	37.6	0.5	38.1	14.3	45.8	22.7
2244x, 2245x	0	100	53.7	27.50	21.5	88.0	14.4	18.0	3.8	21.8	..	15.7	..
Kraft-Type Pulps													
2230x, 2231x	100	0	39.1	20.00	15.6	79.7	10.4	48.5	0.4	48.9	23.0	49.1	23.5
2234x, 2235x	92	8	39.1	20.00	15.6	83.7	10.5	46.5	0.4	46.9	18.1	42.8	27.0
2236x, 2237x	84	16	39.1	20.00	15.6	87.6	10.7	44.5	0.8	45.3	14.6	31.8	28.7
2238x, 2239x	76	24	39.1	20.00	15.6	88.8	10.8	42.6	1.1	43.7	13.2	24.6	31.3
2232x, 2233x, 2240x	0	100	39.1	20.00	15.6	97.1	12.1	14.9	19.4	34.3	..	..	..
Sulphate Semichemical Pulps													
2258x, 2261x, 2265x	100	0	26.9	13.75	10.7	89.0	7.7	..	..	59.2	..	..	..
2262x, 2266x	92	8	26.9	13.75	10.7	92.5	7.8	..	..	61.1	..	..	..
2263x	84	16	26.9	13.75	10.7	93.9	7.7	..	..	60.9	..	..	..
2259x, 2267x	76	24	26.9	13.75	10.7	96.5	7.8	..	..	60.3	..	..	..
2260x, 2264x	0	100	26.9	13.75	10.7	100.0	9.7	..	..	42.0	..	..	..

[a] The cooking conditions other than those tabulated were: weight of moisture-free material, 4 pounds; maximum temperature, 170°C.; temperature-increase period, 1.5 hours; duration at maximum temperature, 1.5 hours; sulphidity (based on active alkali), 30%; liquor-wood ratio (including moisture in chips), 4 to 1.

[b] Pulp bleached with 8.75% chlorine.

of the mixture caused a uniform decrease in pulp yield and an increase in chemical consumption. If 10% of bark by weight is considered the average amount of pine pulpwood, the use of this quantity of bark in the mixture charged to the digester would cause a 2.5% reduction in pulp yield on the basis of the mixture of wood and bark. This reduction in yield on a pulp basis would be about 5%. An unpeeled cord of wood with 10% bark, however, would produce 5.4% more pulp than the bark-free wood obtained from this cord.

These pulp-yield figures are very significant from a wood-utilization standpoint. They mean that the fiber yield per cord of rough pulpwood would be increased by 5.4% by not removing the bark.

On the other hand, material containing 10% of bark consumed 5% more chemicals than bark-free wood. Compared to pulp from bark-free wood, this pulp has a permanganate number higher by 4 units and a little darker color, but it differs relatively little in chemical purity except for a higher lignin content.

The pulp mixtures containing 8, 16, and 24% of bark had higher holocellulose and alpha-cellulose contents and lower lignin contents than predicted from data for pulps of wood or bark alone (Fig. 3). Pentosan and hemicellulose contents were close to those of pulps produced from wood alone. These results indicate that bark has a less harmful effect on kraft-pulp quality than would be expected from the chemical composition of bark pulp.

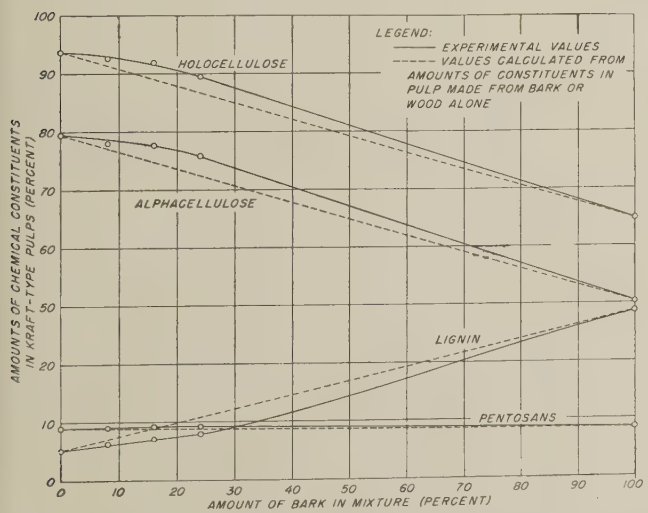

Fig. 3. Relation between chemical constituents in kraft-type pulps and amount of bark in wood-bark mixtures used to make pulp

The most outstanding result in production of kraft pulps from wood and bark mixtures was that pulp strength was not seriously affected when as much as 16% of bark was present. Even with 24% of bark present, the bursting strength of the pulp was only reduced by 8%, the tensile strength by 11%, and the folding endurance by 18%, and the tearing strength remained unchanged. The retention of strength by pulps containing bark is contrary to prevailing opinion and is much more favorable to bark utilization in pulping than would be expected from the extremely weak pulps obtained from bark alone.

Bleaching Sulphate Pulps

The effect of bark in the production of sulphate pulps of bleaching grade was essentially the same as that found for the kraft pulps. Of great importance, however, were the increase in the permanganate number from 16 to 23, the increase in lignin content from 3.3 to 4.1%, the decrease in the brightness of the unbleached pulp, and the marked decrease in the brightness of the pulp after a one-stage bleach test with increase in the amount of bark from 0 to 24% (Table III and Fig. 4). The pulps bleached with greater difficulty than was indicated by their lignin contents and permanganate numbers.

These results indicate that bark, even in small amounts, is very detrimental to the bleaching properties of pine sulphate pulp and that it should be removed entirely from the pulpwood used in this type of pulp.

Sulphate Semichemical Pulps

Sulphate semichemical pulps were made in the 60%

Table III. Chemical Constituents and Strength Properties[a] of Sulphate Pulps Made from Shortleaf Pinewood, Bark, and Wood-Bark Mixtures

Digestion no.	Materials charged, % Wood	Materials charged, % Bark	Chemical constituents, % Holocellulose	Chemical constituents, % Alphacellulose	Chemical constituents, % Lignin	Chemical constituents, % Total pentosans	Duration of beating to freeness of [b], min. 450 ml.	Duration of beating to freeness of [b], min. 250 ml.	Bursting strength at freeness of [b], pt. per lb.[c] 450 ml.	Bursting strength at freeness of [b], pt. per lb.[c] 250 ml.	Tearing strength at freeness of [b], g./lb.[c] 450 ml.	Tearing strength at freeness of [b], g./lb.[c] 250 ml.	Tensile strength, breaking length at freeness of [b], m 450 ml.	Tensile strength, breaking length at freeness of [b], m 250 ml.	Folding endurance of test sheet at freeness of [b], double folds 450 ml.	Folding endurance of test sheet at freeness of [b], double folds 250 ml.	Test sheet density at freeness of [b], g./cc. 450 ml.	Test sheet density at freeness of [b], g./cc. 250 ml.
Bleaching Sulphate Pulps																		
2241x, 2242x	100	0	96.5	81.0	3.3	6.3	42	60	1.00	1.14	2.02	1.66	8800	8800	440	535	0.69	0.73
2243x, 2246x	92	8	94.7	83.3	3.4	7.1	35	52	1.06	1.15	1.97	1.62	8200	8800	400	520	0.68	0.73
2247x, 2248x	84	16	93.8	84.8	3.9	7.2	32	49	1.02	1.11	2.05	1.64	7850	8600	390	540	0.67	0.73
2249x, 2250x	76	24	94.4	83.5	4.1	7.6	30	45	0.99	1.09	1.99	1.73	7700	8400	330	410	0.61	0.68
2244x, 2245x	0	100	79.3	63.7	13.3	9.2	..	3	..	0.19	..	0.21	..	2200	..	1	..	0.44
Kraft-Type Pulps																		
2230x, 2231x	100	0	93.7	79.4	5.1	9.0	46	59	1.12	1.22	2.17	2.02	8700	9000	420	460	0.68	0.70
2234x, 2235x	92	8	92.6	78.0	6.2	9.0	44	62	1.13	1.22	2.22	1.83	7800	8700	360	480	0.65	0.69
2236x, 2237x	84	16	91.8	77.7	7.0	9.2	46	63	1.07	1.22	2.15	1.85	6900	8300	400	570	0.63	0.67
2238x, 2239x	76	24	89.4	75.8	7.9	9.2	45	65	1.05	1.10	2.26	1.85	7400	8400	300	420	0.59	0.64
2232x, 2233x, 2240x	0	100	64.8	50.2	28.6	8.4	..	5[d]	..	0.13[d]	..	0.18[d]	..	1705[d]	..	0[d]	..	0.39[d]
Sulphate Semichemical Pulps																		
2258x, 2261x, 2265x	100	0	81.9	69.1	15.5	9.4	30	50	0.72	0.85	2.05	1.68	5750	6400	140	185	0.52	0.60
2262x, 2266x	92	8	77.5	65.1	19.4	9.1	25	40	0.60	0.73	1.87	1.57	4900	5500	80	110	0.49	0.55
2263x	84	16	74.9	61.2	22.6	9.3	12	28	0.53	0.59	1.75	1.41	4400	4900	50	77	0.47	0.52
2259x, 2267x	76	24	70.3	57.5	24.1	8.9	8	24	0.45	0.50	1.52	1.32	3900	4600	32	47	0.43	0.50
2260x, 2264x	0	100	54.3	41.9	37.3	7.1	..	0[e]	..	0.08[e]	..	0.20[e]	..	840[e]	..	0[e]	..	0.33[e]

[a] Interpolated test-beater strength data on unbleached pulp. Test sheets were conditioned and tested at 23°C. and 50% relative humidity.
[b] Canadian-Standard freeness.
[c] Weight of ream of paper containing 500 sheets, each 25 by 40 inches in size.
[d] At a freeness of 140 ml.
[e] At a freeness of 65 ml.

yield range from the various mixtures of wood and bark. The softened chips resulting from the partial chemical pulping were fiberized in an attrition mill to separate the fibers and produce a pulp suitable for liner board. The effect of bark in semichemical pulping was somewhat different than that found in producing kraft and bleaching pulps. An increase in bark content caused an increase in the chemical consumed as with the other pulping methods, but caused a decrease in pulp strength with no appreciable change in pulp yield (Tables II and III). The yield from bark alone, however, was nearly 30% lower than that from wood alone.

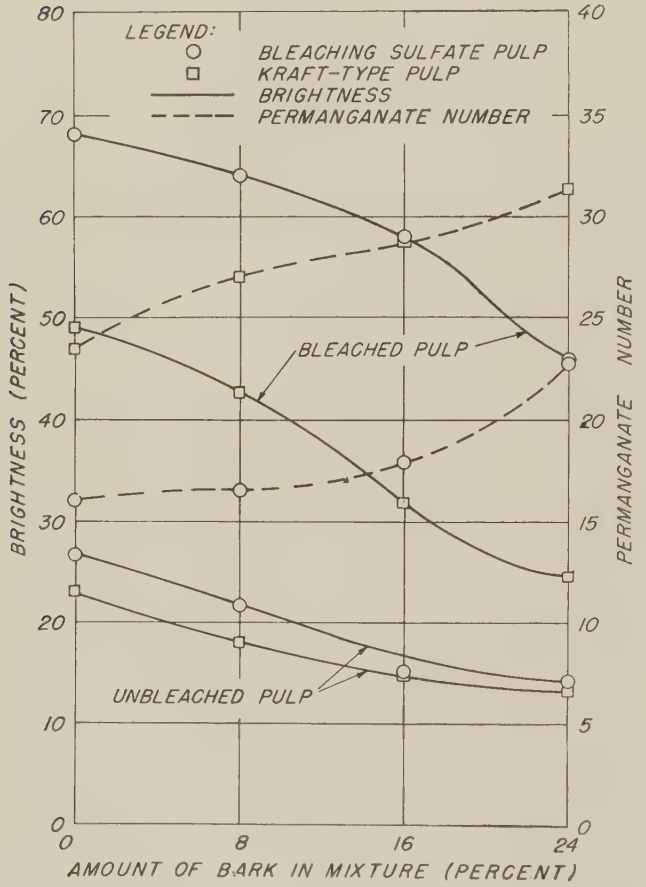

Fig. 4. Effect of bark on permanganate number and brightness (before and after a one-stage bleach test with 8.75% chlorine) of kraft-type and bleaching sulphate pulps

The strength of the semichemical pulps decreased appreciably with increasing amounts of bark. Pulp with 16% of bark had 30% less bursting strength and 15% less tearing resistance than the pulp from the wood alone. The test sheets made from the bark pulps were less dense than that from the wood alone. The comparatively lower strength of the pulps with bark appears serious enough to prevent their use in products having the highest strength requirements.

The lignin contents of the pulps from the wood-bark mixtures were considerably higher and the cellulose contents were lower than the values calculated from the analyses of the pulps made from wood and bark alone. For example, the substitution of 24% of bark caused an increase in lignin of 55%, instead of the calculated 35%, and a decrease in holocellulose of 14%, instead of the calculated 8%. Under the conditions of partial pulping, the bark may have been selectively pulped to a greater degree than the wood. This would explain the constant yield and the decrease in strength with increase in bark present, although it is contrary to experience in semichemical and chemical pulping of mixtures of woods having widely different pulping characteristics.

Because of the increased yield of pulp from a cord of rough wood, considerable advantage could be realized by not removing the bark from pulpwood used in sulphate semichemical pulping.

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Flash Drying of Lime Kiln Feed

HENRY VRANIAN and ROBERT D. NICKERSON

Flash drying of calcium carbonate lime kiln sludge is used for the first time to supplement the capacity of an existing rotary kiln in a pulp mill. The carbonate product is delivered from the drier to the kiln at a controlled moisture content. Most of the former drying zone in the kiln is now available for calcining resulting in increased lime-burning capacity. The heat for the drier is drawn from the plenum chamber of the lime kiln stack. Under the present conditions the drier does not use all the available heat and some is bypassed to the stack. As a result of drying the lime-burning capacity has increased by 40% . . . fuel consumption reduced by 38%, and make-up lime reduced by 37%. Use of a secondary dust collector is considered advisable and should reduce the make-up lime further.

THE purpose of this paper is to report on the use of a flash drier in conjunction with a rotary kiln used for reburning pulp mill lime mud. This combination of a flash drier with a rotary kiln is in operation at The Chesapeake Corp. of Virginia at West Point, Va. It is the first successful installation of its type.

Because of increasing the kraft pulp production at The Chesapeake Corp. it became necessary to expand the lime recovery capacity. The present kiln, 9 by 120 feet, was installed in 1935, when the pulp production was 270 air-dry tons per day with 340 pounds of lime used per ton of pulp. Since then, pulp production has increased to a maximum of 570 air-dry tons per day. With the same lime recovery equipment the losses and fuel costs became increasingly greater so that the need for additional capacity became urgent.

An additional kiln would have had to be placed remote from the existing recovery facilities because of space limitations. This, in turn, would have resulted in additional labor and more equipment for returning the lime to the present causticizing system. The rather large first cost of an installed kiln was an important factor also, and as a result, the following possibility was considered.

The kiln at The Chesapeake Corp. has a natural draft hood at the firing end and has never been equipped with a chain section or lime cooler. As the need for greater capacity mounted it became evident that a considerable amount of heat was being wasted at the kiln stack and calculations showed that it was sufficient to dry the calcium carbonate mud from the vacuum filter. It was reasoned that with a dry feed to the kiln much of the drying zone would become available for calcining, thereby increasing the kiln capacity.

Further investigation indicated that the gas temperatures leaving such a system would be lower so that improved heat economy would result in lower fuel consumption.

The principle of flash drying had been used in other industries for products of this nature and appeared feasible for this application. A full-scale system, designed to fit in the existing space, was installed and began operating in the spring of 1951.

Since then, the system has been on the line with a dependable performance record.

RESULTS

As a result, the capacity of the lime recovery system has been increased, the fuel consumption lowered, and the lime quality unchanged. Comparing a recent 20-week period with the 1950 average, the following savings have been realized: (Table I) purchased lime reduced from 106 to 67 pounds CaO per air-dry ton of pulp while the reburned lime capacity increased from 57 to 80 tons per day; also, Bunker C fuel consumption decreased from 108 to 67 gallons per ton lime.

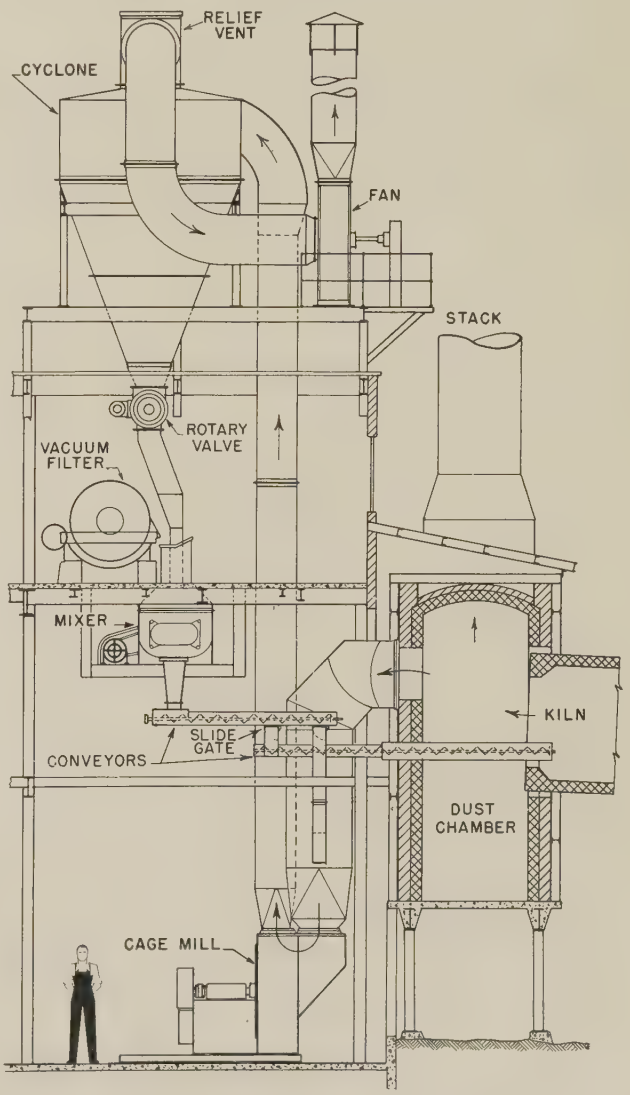

Fig. 1. Flash drier for lime kiln feed at Chesapeake Corp. Arrows indicate direction of gas flow

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Table I. Comparison of Performance Before (1949–50) and After Installation of Flash Drier (20 Weeks 1951)

	1949	*1950*	*1951, 20 weeks*
Make-up lime, lb./air-dry ton	100	106	67
Reburned lime, tons/day	52	57	80
Bunker C fuel oil, gal./ton lime	94	108	67

In the same interval there was an increased load, due to a pulp production gain, of 19%. The previous quality standard of 87 to 90% available CaO is readily maintained.

The present reburning capacity of 80 tons per day is the over-all average without allowance for downtime. In the 20 weeks' period there was a total of 7% downtime for various reasons, and with this allowed, the reburning rate is 86 tons per day.

The reduction in lime make-up is principally due to the increased reburning capacity of the kiln which eliminates most of the mud wastage previously experienced. It is believed that the make-up of 67 pounds can be further reduced (1) by the use of a secondary dust collector for the flash drier and the present bypass stack and (2) by certain improvements to the original caustic equipment.

Reported experience at other pulp mills using wet collectors on exhaust gases from conventional kilns has indicated the possibility of greatly reducing carbonate losses by this means.

As more experience is gained, it is reasonable to expect that the thermal efficiency of the combination will be increased, thus, further reducing the oil per ton of reburned lime.

DESIGN AND PRINCIPLE

The principle of the flash drying system is well known and has been described in previous papers (*1*, *2*). However, this particular application is unique and its requirements are distinctly unusual. Also this type of drying system is relatively new in the pulp mill field, and a brief description of its principles and design is in order.

Flash drying may be defined as the practically instantaneous removal of moisture from materials by the application of heat in a turbulent stream of hot air.

Since maximum agitation is fundamental to instantaneous drying, special provision is made to secure it. This is accomplished in a cage mill which consists of a special rotor enclosed in a housing (see Fig. 2). The wet material and the hot gases are brought into intimate contact and the water vapor blanket on each particle is continuously removed by the agitation provided.

Actual design of the flash drier is based upon the characteristics of the material to be dried and known data obtained from tests on plant size equipment. The inlet drying temperature which may be permitted together with the probable vent temperature, the particle size, density, final moisture, and color of the product are generally determined by test. Then the equipment is sized by calculating the weight of gas required for drying using a simple heat balance and the laws of airflow and fan design.

The flow of material and gases through the system is as follows:

The vacuum filter discharges its mud to the mixer (see Fig. 1) where it is blended with all of the dried carbonate discharged from the flash drying system. From the mixer, a screw conveyor moves the material over an adjustable slide gate through which the proper proportion of damp material drops to the kiln feed screw. The remainder goes to a chute and then to the cage mill shown. Here, the drying is done by the cage mill agitating the carbonate in the hot gases drawn from the kiln as described above.

Both the gases and the carbonate are carried upward from the cage mill through the connecting duct work to the cyclone. Here, the now dried carbonate is separated out by centrifugal action and discharged through a rotary valve to the mixer.

The spent drying gases are drawn from the cyclone by the vent fan and discharged to mixer.

This fan was selected to allow the use of a secondary dust collector should it be desirable or necessary. Operating experience at Chesapeake indicates it to be necessary from the standpoint of nuisance and reduction of lime losses. Other pulp mills with units under contract are to be equipped with secondary collectors.

MAINTENANCE

Inspection of the flash drier after 6 months of continuous operation showed that the machinery items were in excellent condition. However, three points in the duct work had excessive wear due to abrasion. These points were originally constructed of mild steel. They are to be replaced with resistant materials commonly used for such service in flash driers for drying coal and sewage sludge.

OPERATION

No additional personnel is required to operate the added equipment since it is attended by the existing caustic operators. They have found no difficulty in starting or stopping the drying operation. The kiln operation, according to the operators, is not impaired by this addition. In fact, they report the operation has slightly improved presumably due to a more uniform feed.

The vacuum filter averages 30 to 35% moisture in the mud feed to the flash drier. It is interesting to note that prior to this installation of the flash drier, the vacuum filter cake averaged 40 to 45% moisture. The kiln feed averaged 8 to 10% moisture. The flash-dried product averages 1% moisture.

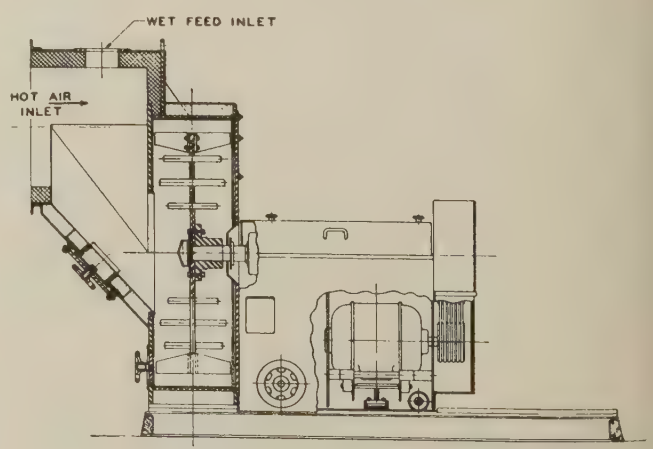

Fig. 2. C-E Raymond flash drier cage mill showing rotor

The vent temperature from the flash drier is 150°F. This is controlled automatically by regulating the temperature of the hot waste gases from the kiln which enter the drying system. The amount and temperature of these gases are dependent upon the evaporative load at any particular time. The hot gases not used are bypassed and provide a substantial margin in case the oil consumption now required for calcining is reduced. The calcining operation now dictates the amount of fuel burned.

One of the essential requirements is that the dried carbonate be fed to the kiln in such a state that it will remain in the kiln and not "blow back." This is a vital part of the drying operation and is accomplished by controlling the moisture content of the carbonate fed to the kiln as follows:

Since the operator has only minor control of the amount of cake produced by the filter, it is obvious that control of the moisture in the kiln feed must be regulated by the slide gate in the mixer discharge screw. If the kiln feed is too dry the gate is opened more thus reducing the amount of carbonate going to the flash drier. This reduces the dried product discharged to the mixer thus increasing the per cent moisture in the mix.

The reverse of this, partial closing of the gate, takes care of excess moisture in the kiln feed. In practice, the gate remains unchanged for days.

The kiln operator burns sufficient oil to obtain calcination without regard to drying requirements. To date, this has resulted in a satisfactory product but with an excess of hot gases which are wasted. With further experience, it may be possible to further reduce the oil consumption to the point that most of the hot gases will be used in the flash drier and at the same time secure the necessary calcining action.

The ultimate capacity of the combination has not yet been determined. The operating rate of the combination is 86 tons per day, and the flash drier is capable of a one third increase in its present rate provided the kiln can calcine that much.

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A Note on the Theory of Fiber-Fiber Bonding in Paper; the Influence on Paper Strength of Drying by Sublimation

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It is generally appreciated that the strength of ordinary paper resides primarily in the bonds between the fibers. An increase in beating action enhances tensile strength and bursting strength through promotion of more extensive bonding between the fibers which, in turn, may be attributed to increase in fibrillation and softness of the fibers; it is also possible, many believe, that the brushing action of beating augments the specific cohesion between fibers. Ultimately, as the degree of beating is extended to higher values, the strength of the paper attains a maximum value, and further increments in the degree of beating cause the strength to decrease. In this regime of beating, fiber-fiber bonding is almost at its maximum possible value (as evidenced by high density and low optical scattering) and the fibers are weakened by fibrillation and other effects of the beating action.

In order that fiber-fiber bonds may be formed, elements of contiguous fibers must be brought into essentially molecular contact. Wet pressing provides the mechanical stresses for close contact between fibers, and it is known that wet pressing has a strong influence on the strength of the dry paper. However, wet pressing alone would seem to be incapable of producing the relatively strong bonds existing in the dry paper. Immediately after pressing, the wet sheet has very low strength; furthermore, the surfaces of the fibers are undoubtedly covered with layers of associated water many molecules thick, which prevent the creation of strong, permanent bonds. Wet pressing deforms the fibers of the sheet and creates an environment in which the fibers are in close contiguity. Because of the slight resilience of the wet fibers, a "spring-back" occurs after pressing, and fiber elements which were in close contact during pressing will generally separate somewhat.

Nearly 20 years ago, Campbell (*1*) published a theory which affords a plausible explanation for the bringing together of fiber elements to a degree of proximity permitting the force fields of the elements to react and form bonds. During the drying of the sheet (when the moisture content is below the fiber saturation point), water is located in the microcapillaries of the fibers and between fibers in the zones of near contact. According to Campbell's theory, surface tension pulls fibers and fibrils together during the drying process; of course, this action of surface tension will prevail only at those points in the sheet where the stiffness of the fiber mesh and the distance of approach of fiber elements are sufficiently small for contiguous fiber elements to yield under the stress caused by surface tension and to move together. Presumably the number of such points in a paper sheet would be very large, but would depend upon such factors as the nature of the fiber, the kind and degree of beating action, the nature

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of felting together of the fibers during formation of the sheet, and the pressure and duration of pressure during the wet pressing. As drying proceeds, the films of water between contiguous fiber elements diminish in thickness and, in consequence, the *tension* stress in the water increases, thus causing the elements to be brought together with increasing force. If x is the thickness of film of a liquid of surface tension λ between two flat surfaces, and if the liquid wets the surfaces but does not extend out to the edges of the surfaces, then the tension stress in the liquid is $2\lambda/x$ dynes per sq. cm. (The magnitude of this stress may be very large, as the following example will show: if $\lambda = 70$ dynes per cm. and $x = 70$ A., then the tension stress should be $2 \times 70/(70 \times 10^{-8}) = 2 \times 10^{8}$ dynes per sq. cm. = 200 atmospheres.) Campbell points out that the surface tension of the water in a thin film may be different from that of water in bulk and calculates the order of magnitude of the stress in the water from the formula (developed with the aid of thermodynamics):

$$P = (1.95 \times 10^{5}\, T\sigma g/M) \log_{10} (p_0/p) \text{ dynes/sq. cm.}$$

in which P is the hydrostatic pressure in the liquid, T is the absolute temperature, σ is the density of the liquid, g is the acceleration of a freely falling body, M is the molecular weight of the liquid, p_0 is the vapor pressure of the liquid when its hydrostatic pressure is P, and p is the pressure of saturated vapor of the liquid. Campbell shows that when a sheet of paper is in equilibrium with water vapor at 0.9 saturation (90% relative humidity), the tension stress in the water in the sheet should be about 145 atmospheres; the tension stress increases with increasing dryness.

The purpose of this article is to present evidence supporting Campbell's theory of the action of internal tension in water on the formation of fiber-fiber bonds during the drying of paper.* If the fiber-fiber bonds are established during the pressing operation and strengthened by removal of water *without appreciable contribution from internal tension, then a sheet of virtually normal strength should be obtained by a drying process in which water is removed in such a manner that internal tension is restrained from pulling fiber elements together into very close contact.* Drying by sublimation, that is, removing water in the vapor phase from the solid, or ice, phase, should provide a test of the theory. Apparent defects of this test of the theory are: (1) Water expands on freezing and, hence, fiber elements would be moved apart. (2) The formation of ice might be expected to weaken the fibers. These effects can be allowed for through the use of suitable controls.

In the first experiment on the effect of drying by sublimation, three handsheets were prepared from well-beaten unbleached sulphite pulp. In order that any effect of the initial action of surface tension might be emphasized, the test sheets were not pressed; the only pressing action which they received was that produced by the couching roll of the British sheetmaking equipment. They were couched on blotters, and left on the blotters until dry. One sheet, labeled RT, was permitted to air dry at room temperature. The other two were labeled, respectively, LT and LT-C (low temperature and low temperature control). These were placed between sheets of moistureproof paper so that they could be transported to a cold box without loss of water by drying. Placed in a cold box at −40°C., they were rapidly frozen to minimize fiber damage resulting from ice crystal growth. One hour after freezing, the control sheet (LT-C) was removed from the cold box and allowed to thaw without loss of moisture, following which the moistureproof sheets were removed and the control sheet was permitted to air dry. Sublimation of water from sheet LT was, of course, very slow at −40°C. and, subsequently, this sheet was transferred (without appreciable temperature rise) to a cold box in which the temperature was controlled at a value not far below the freezing point, −6°C. Sublimed moisture condensed upon the refrigerated copper coil in this box. After 2 days at −6°C., the sheet presented an ice-free appearance, and could be easily peeled from the blotter. After 4 days, the moisture content had fallen to 10% (as measured by weighings on a small specimen cut from the sheet and introduced in a weighing bottle momentarily placed in the cold box). On the sixth day, the moisture content was 9%. The specimen was removed between cold glass plates on the seventh day, and permitted to warm to room temperature before air drying was initiated by removal of the glass plates. (Presumably, some of the bonds would be strengthened by further drying from a moisture content in the neighborhood of 9%, but the estimated time required to dry the sheet to about 5% moisture content at −6°C. seemed excessive in view of the preliminary nature of this experiment.)

All three handsheets were conditioned at 73°F. and 50% relative humidity prior to testing. Data on tensile strength and optical properties of interest are presented in Table I. It will be seen that the strength of the sheet dried by sublimation (LT) was about 40% of that of the normal sheet (RT) and very substantially lower than the strength of the control sheet (LT-C). The low strength and high opacity and brightness of the sheet dried by sublimation may be clearly interpreted as evidence for low degree of bonding between the fibers in that sheet. Because of the nonlinear relationships between optical scattering coefficient and optical properties, it may be said that the difference between the bonded areas in LT and LT-C is substantially greater than would superficially be indicated by the opacity values (0.94 and 0.90). The sheet dried by sublimation possessed all of the subjective qualities of paper made with unbeaten pulp—it felt soft, bulky, and flexible. The properties of LT-C were roughly intermediate to those of LT and RT; this is attributed to the expansion of water on freezing. A portion of the difference in strength between RT and LT is, of course, attributable to this effect, but there seems to be little doubt that much of the decrement in strength is due to the elimination of the influence of internal tension of water during the drying process.

Table I

Sheet designation	Basis weight, lb. 25 × 40—500	Tensile, lb./in.	Opacity, $R_0/R_{.89}$	Brightness, %
LT	47.4	17.8	0.94	63.4
LT-C	47.4	31.2	0.90	57.1
RT	47.3	43.0	0.73	44.0

In a second experiment, an attempt was made to subject a control sheet to low temperature-time conditions

* The work reported in this article was conducted in 1947.

similar to those of the LT sheet, with the LT-C sheet confined in a Pliofilm envelope to restrain drying. It was also hoped, in this experiment, to obtain a rough measure of the extent of fiber damage caused by the formation of ice in the sheets. Three handsheets were prepared from unbleached sulphite pulp beaten to a freeness of 690 ml. S.-R. in a Valley laboratory beater. These sheets, labeled as before, were subjected to wet pressing. The LT and LT-C sheets were placed in Pliofilm envelopes and quickly frozen at −40°C. A few hours later, they were transferred, without substantial warming, to the cold box in which the temperature was controlled at −5°C., and the Pliofilm envelope was removed from the LT sheet so that sublimation could proceed. Eleven days later, when the moisture content was below 9% (as judged from the first experiment) and was probably in the neighborhood of 8%, the sheets were removed from the box. It was observed that a substantial amount of the water in the LT-C sheet had condensed upon the Pliofilm envelope, so that this sheet had dried partially by sublimation in spite of the moistureproof envelope. The Pliofilm envelope was not removed until the envelope and the specimen which it contained had warmed to room temperature and the moisture had transferred from the envelope to the specimen sufficiently to give the specimen a wet appearance. The LT-C sheet was permitted to air dry at room temperature from this condition.

After conditioning at 73°F. and 50% relative humidity, the data presented in Table II were obtained. The data corroborate the results and conclusions of the first experiment (Table I). Again, the strength of the sheet dried by sublimation was about 40% of that of the sheet dried at room temperature. Because of the previously described difficulty encountered with the control sheet LT-C, an accurate measure of the effect of expansion of water on freezing cannot be made. However, the result for LT-C shows that prolonged exposure of a sheet to −5°C. will not cause the full decrement in strength exhibited by LT.

Table II

Sheet designation	*Basis weight, lb. 25 × 40—500*	*Tensile, lb./in.*
LT	44.6	8.7
LT-C	44.7	12.1
RT	44.6	22.3

An estimate of the magnitude of fiber damage caused by the formation of ice in a sheet (under the stated conditions of quickfreezing followed by a prolonged interval at −5°C.) was obtained by forming handsheets from the scraps of the test sheets. The scraps from the sheets were soaked in distilled water overnight, defibered in a malted milk mixer, and formed into handsheets, using, again, the British sheetmaking equipment. As shown in Table III, this operation involved a loss of only 2% of the fiber of each sheet. The tensile strengths of the three sheets air dried at room temperature are of the same order of magnitude. Most of the drop in strength from 22.3 pounds per inch—the original strength of sheet RT—is attributed to the well-known effect on strength of drying of fiber. The small but probably real difference between the strength of sheet RT-RT and the other two sheets is probably attributable to fiber damage or other effects of ice formation. The data presented in Table III show that any loss in strength attributable to ice formation is relatively small as compared with the differences in strength between LT and LT-C sheets (Tables I and II).

Table III

Sheet designation	*Basis weight, lb. 25 × 40—500*	*Tensile, lb./in.*
LT-RT	43.6	10.2
LT-C–RT	43.8	10.5
RT-RT	43.9	12.3

The fiber elements in sheets formed in liquids other than water should be brought together by internal tension during the drying. However, the intimate bringing together of fiber elements would appear to be a necessary but not sufficient condition for the formation of strong bonds in the dry paper. Kress and Bialkowsky (*2*) have shown that liquids which cause cellulose fibers to swell, such as water, ethylene glycol, and formamide, promote strong bonds between fibers, whereas beating in higher alcohols and hydrocarbons results in "very free and fluffy stock" (low degree of bonding between the fibers). Undoubtedly the lower bonding strength resulting from treatment with a liquid of the latter class is due to a combination of possible effects, such as (1) the existence of residual layers on the fibers of the sorbed liquid which inhibit the formation of strong bonds, (2) failure of the liquid to swell and soften the fibers, and (3) absence, in the presence of the liquid, of surface molecular modification on the cellulose required for the formation of strong bonds. In the work reported in the present article, the fibers were beaten and formed in water, so that normal swelling and surface modification of the fibers occurred, and any bond-inhibiting effects caused by foreign liquids were absent. It is clear, then, that a portion of the observed decrement in strength in the LT sheets is attributable to the absence, during drying, of a pulling together of fiber elements induced by internal tension in the water.

Future research will be based on a number of questions which suggest themselves. For example, the connection between the internal tension effect and wet pressing should be thoroughly explored; a range of pressure-time schedules in wet pressing should be examined; the effect of "spring-back" of the wet sheet after pressing should be studied; the work should include observations on the effect of drying (at both room and low temperature) while the sheet is under a steady pressing load. The importance of surface tension of the water *in the sheet* (at the temperature of drying) on the strength of the finished paper should be examined, with particular reference to the influence on surface tension of sheet constituents.

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Waste Water Disposal Program, Scott Paper Co., Chester, Pa.

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Events leading toward installation of a waste water disposal plant at the Chester, Pa., plant of Scott Paper Co, are covered to show how the present plant was determined. Preliminary to filing plans with the State Authorities for a complete treatment plant for waste waters discharged into the Delaware River, Scott Paper Co. conducted tests on a standard full size precoat type filter to obtain sufficient data for the design of a waste water disposal plant. Tests showed for example that the capacity of a Waco filter operating at top speed on paper machine white water having a fiber content of 8 pounds per thousand gallons was 600 g.p.m. but the capacity increased to 1400 g.p.m. when operating on lean waste water at 1.5 pounds per thousand gallons. Using the test results as a guide a complete waste water disposal plant was designed and built using Waco filters, and including a novel water collection system. The plant operation is completely automatic with a recovery of over 90% of the incoming fiber. The collection system consists of an elevated open flume which receives white water from numerous pipe lines fed by individual pumps. The flume conveys the water to a 100,000-gallon collector tank which serves to even out surges. Paper machine furnish is used for precoat and together with the recovered fiber is returned for reutilization in the papermaking process. Actual operating records during the first 6 months show that filter performance has lived up to predictions based upon pilot plant studies.

In March of 1950, a waste water disposal plant was placed in operation at Scott Paper Co., Chester, Pa., which represented the culmination of a long period of planning and testing in the direction of reducing pollution of the Delaware River. It may be said that a substantial improvement has been made in Scott's waste water disposal situation at this mill.

In order to have a better understanding of the progress made on this important paper mill problem it is necessary to dwell momentarily at least on the circumstances and events that led to the final selection of waste control equipment.

EARLY EVALUATION OF THE MILL EFFLUENT

During the Fall of 1947, the Chester Mill of Scott Paper Co. received authorization from the Commonwealth of Pennsylvania to start construction of a waste control plant. The stated object of the plant was to reduce the pollutional load of Scott's industrial waste discharged to the Delaware River by at least the equivalent of "primary treatment" as defined by sanitary sewage rules.

It is of particular importance to know that stream pollution control has received much added emphasis since the war. Recent information reveals that 44 of the 48 states have pollution laws of one kind or another, and it is probable that Pennsylvania is conducting one of the most active campaigns to clean up its streams.

On first consideration, the requirements of the State might not appear to present an unduly difficult problem. However, in 1947, the waste water discharged to the Delaware River from the Scott mill at Chester consisted of approximately 7,000,000 gallons per day containing 0.02% of pulp fiber. Accepted methods of removing fibrous material from such an aqueous solution usually entailed chemical treatment of the waste water followed by settling in large size sedimentation basins. The clarified water is then discharged to the stream and sludge recovered from the basin is disposed of by lagooning or reutilization in the papermaking process. For Scott Paper, this method of treatment had serious complications.

1. No land was available around the Chester mill to permit construction of lagoons for sludge disposal.
2. It was not considered practical to reutilize the sludge in the papermaking process because it would contain heavy dosages of alum and activated silica which would be unaccepted in the finished product.
3. No acceptable methods of concentrating the sludge for baling or other disposition were then known to be operating successfully.

With this background, it was difficult to decide upon a suitable approach to this problem. Also, time was of the essence in completing pilot plant work in order that construction could be started. About this time the Waco filter, a new type of precoat filter developed in Scandinavia, was first introduced in the United States and Canada. The filter was originally developed to purify fresh water for municipalities and subsequently was adopted for use in paper mills as a machine saveall.

Experience with this machine in Scandinavia demonstrated its stock recovery efficiency to be extremely high and capacity to vary considerably depending upon weight and characteristics of both precoat and recovered stocks. In addition, this filter appeared to have certain features which made it advantageous as a possible solution for the waste control problem at Chester.

1. Chemicals for coagulation and settling such as are necessary for sedimentation units were not required.
2. The possibility for using regular paper machine furnish for mat stock seemed excellent.
3. As neither vacuum nor pressure was utilized to increase capacity, the character of the effluent would probably be good.

Preliminary experiments were conducted employing a leaf tester of the type recommended by the designer. The results were most encouraging. As a matter of interest, the performance actually obtained correlated very closely with the leaf tests. The characteristics of

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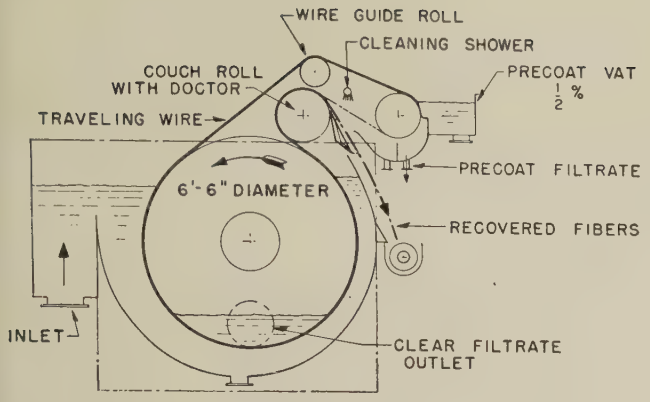

Fig. 1. Diagram of WACO filter showing principal parts

the effluent matched results predicted from the performance of filters operating in several Scandinavian mills. Results looked promising and it was decided to install a full size filter and conduct pilot plant studies. At this point, it should be stated that during preliminary and pilot plant work, the Commonwealth represented by the Sanitary Water Board and its district engineer was cooperative and displayed considerable interest in the project.

DESCRIPTION OF WACO FILTER

The general configuration of this type of precoat filter is shown by Fig. 1. The principal parts of the Waco filter consist of:

A large open vat containing a cylinder 14 feet long and 6 feet 6 inches in diameter constructed of steel ribs fitted with bronze tips. This cylinder is provided with a variable speed drive motor having a speed range of 10 to 40 f.p.m.

A bronze-covered couch roll to provide a means for removing stock from the traveling wire face.

A bronze-covered guide roll equipped with an automatic guide at one end and a manual guide at the other.

A precoat vat containing a small open cylinder with top feed headbox. It is on this cylinder that the precoat is formed much in the manner of a decker. Water from furnish pumped to the headbox drains through the wire leaving a thin precoat on the traveling wire. The headbox is supplied with furnish by a circulating system much like that used on a conventional paper machine.

The four rolls described above are wrapped by a traveling bronze wire 45 by 50 mesh. A precoat is formed on the wire and carried over the guide roll into the main vat containing water to be filtered. Untreated water filters through the pulp mat depositing fiber on the precoat and drains away to the sewer or is reutilized in process work. The precoat together with recovered fiber is carried over the couch roll which removes the fiber from the wire, and a doctor blade serves to remove the precoat from the couch roll. The combined mat and recovered stock are then diluted and pumped to the pulp preparation system for reutilization. In brief, it may be said that this type of precoat filter effectively brings to production the laboratory process of filtration through filter paper.

Table I. Typical Waco Filter Performance on Waste Water

Wire speed, f.p.m.	*Mat stock, lb./sq. ft. wire*	*Influent consistency, lb./1000 gal.*	*B.O.D., p.p.m.*	*Effluent consistency, lb./1000 gal.*	*B.O.D., p.p.m.*	*Capacity, g.p.m.*
10	0.008	1.44	160	0.04	85	475
15	0.014	1.25	130	Nil	76	600
25	0.013	2.00	130	0.18	62	800

PILOT PLANT STUDIES

A full size filter was installed in the Spring of 1948. It was piped to permit operation with six different combinations of mat and unfiltered white water. This was done not only to determine performance of the filters on waste waters, but also to determine its potential as a machine saveall. This discussion will be confined to results obtained using the equivalent of waste water and one type of machine furnish for mat stock. The results obtained are characteristic of the performance with other combinations of mat stock and unfiltered water and they also served as the design basis for the subsequent plant. Characteristic results from these tests are given in Table I for three different speeds.

Throughout the tests the filter performed with remarkable uniformity, the characteristics of the effluent remaining for the most part between 0.10 and 0.20 pound per thousand gallons.

Additional results of the pilot plant tests are best shown by the following curves.

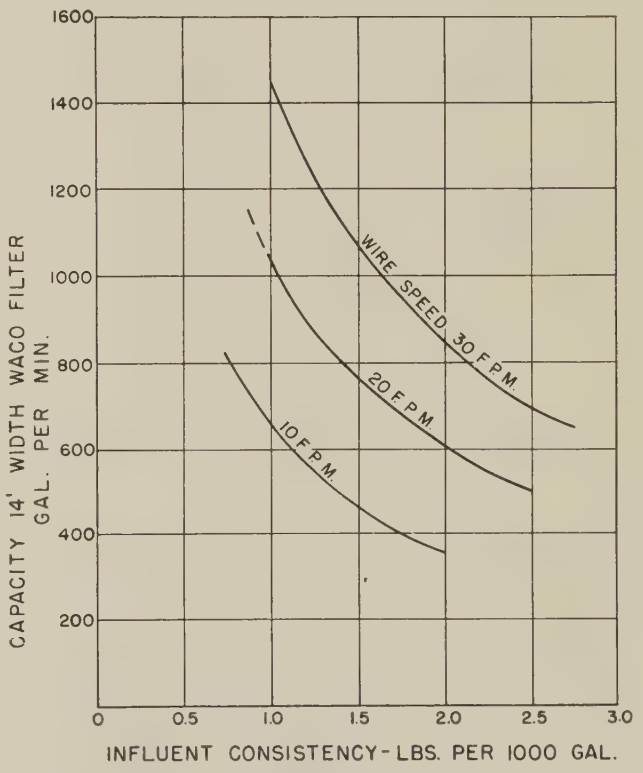

Fig. 2. Capacity of WACO filter vs. consistency of influent waste water

In Fig. 2, the consistency, pounds per 1000 gallons, of the unfiltered water is plotted against the capacity in gallons per minute. Three different wire speeds are plotted as parameters. In this curve it is particularly significant to note that a small change in con-

sistency of the unfiltered water varies the capacity of the filter greatly. Needless to say, this fact makes an accurate study of the actual characteristics of the unfiltered water very necessary.

The consistency of the filtered water in pounds per thousand gallons is plotted (Fig. 3) against the precoat weight in pounds per square foot. It would appear as though little is to be gained by operating a filter with a precoat weight greater than 0.014 pound per square foot. The full scale plant operates in actual practice with a precoat weight ranging from 0.012 to 0.020 pound per square foot. In passing, it is interesting to mention that the clarity of the effluent improves as the percentage of short fiber pulp in the furnish increases. Also, the capacity decreases with increase in short fiber pulps. While not shown in these particular curves, it was found to be the case in other phases of the pilot plant work.

The filter capacity in gallons per minute is plotted (Fig. 4) against wire speed in feet per minute. Consistency of the unfiltered water in pounds per thousand gallons are the parameters. These curves also show the effect of higher proportions of short fiber pulp in the unfiltered water. The dotted lines represent unfiltered water containing a high percentage of long fiber whereas the solid lines represent unfiltered water containing a high percentage of short fiber pulp.

Throughout the pilot plant work periodic B.O.D. tests were made. Generally speaking it was found that by removing most of the solids a B.O.D. reduction of 30 to 50% could be effected. All sampling and testing throughout the pilot plant studies were performed according to regularly accepted practices.

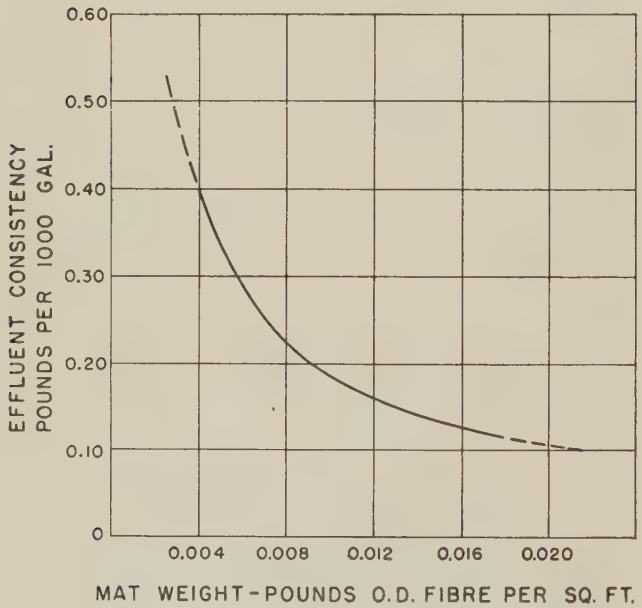

Fig. 3. Effect of precoat weight on clarity of effluent

Some may wonder how well the performance of this type of precoat filter compares with that of a so-called, high-rate flocculator. During two successive summers pilot plant studies were made with this type of equipment and it is particularly interesting to note that the effluents of the two types of apparatus compare favorably.

THE COLLECTOR SYSTEM

At the same time that the performance characteristics of the Waco filter were being determined a study was

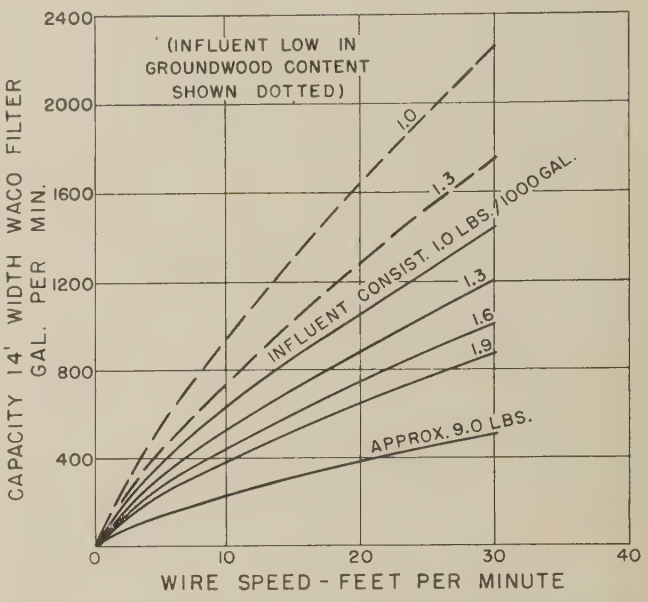

Fig. 4. Capacity of WACO filter vs. wire speed

inaugurated to classify the sources of waste water and estimate the quantity of each being discharged to the sewer. These sources were arranged into two groups. The first and most important group consisted of lean white water which had been through a paper machine saveall. The excess water leaving the saveall seal boxes from each of nine paper machines varied between 100 and 300 g.p.m. and the fiber content was fairly uniform in the range of 1 to 2 pounds per thousand gallons. The excess at each seal box represented a known fiber loss calculated to be no greater than sufficient to purge the white water system so as to avoid high concentrations of dissolved solids. The presence of fiber in the saveall effluent reflected the presence of "fines" which were not retained on the paper machine wet end and also showed that the saveall capacities were somewhat exceeded for best results. The fiber content of this white water was assumed to have value since the water was free from objectionable foreign matter.

The second type of mill effluent was found to be associated with the various drains in the machine room basements. For one reason or another this water was contaminated. While the fiber content was sometimes high caused by machine washups, etc., it was agreed that inclusion of this water in the waste treatment system might introduce material which could not be tolerated. It was believed that a method for disposing of this type of waste water would require development sometime soon and that there was a possibility that these drains might be included in the filtering operation.

The physical location of these sources of waste water presented a formidable problem. Visualize a

series of machine room basements having a total length of over 600 feet with a total of 15 sources of waste water.

The collector system (Fig. 5) as it is called, consists of: an elevated open trough made U-shaped and

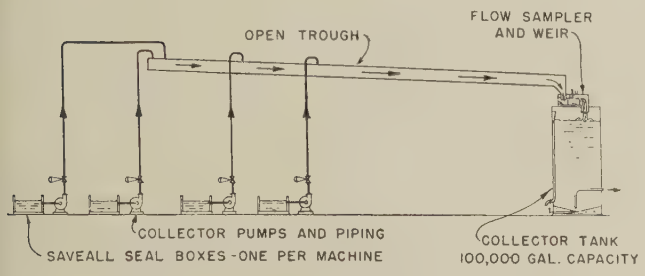
OPEN TROUGH
FLOW SAMPLER AND WEIR
COLLECTOR PUMPS AND PIPING
SAVEALL SEAL BOXES-ONE PER MACHINE
COLLECTOR TANK 100,000 GAL. CAPACITY

Fig. 5. Diagram of collector system

located for the most part on the roof of the mill buildings, 15 collector pumps, and pipe lines all of which discharge waste water into the trough, a 100,000-gallon collector tank and a main weir for flow measurement as well as a flow sampler. The pumps were selected for duplication; only three different sizes were used. The small pumps selected were close-coupled motor pumps because in most locations, space was at a premium.

Each source of water is provided with a level control device which actuates a butterfly control valve in the discharge pipe lines. The collector piping is prefabricated from steel pipe and has a Lithcote lining.

The collector trough is fabricated from steel plate in sections and is also Lithcote lined, its length being over 400 feet. The trough slopes gently toward the collector tank and is designed to handle 8000 g.p.m.

The collector tank (Fig. 6) receives waste water from the trough after the water has been measured over a weir. The large capacity of the collector tank provides a means of equalizing the flow rate and the solids content and contributes a steadying influence on operation. The bottom has a slight cone shape to draw any settled solids toward the center where the drawoff pipe is located. The drawoff line curves upward from the center of the tank thereby leaving any heavy solids in the collector tank which otherwise might damage the wires on the Waco filters. The collector tank has a high level overflow which may be used periodically to flush away floating solid material and to wash the tank walls. The walls of the tank have a Durofilm protective coating.

FULL SCALE WASTE WATER PLANT

The four Waco filters (Fig. 7) are the heart of the waste water disposal plant. The machines were redesigned by Improved Paper Machinery Co. The filter operation in the full scale plant is the same as for the single experimental filter unit described above. But, of course, the auxiliary equipment providing the precoat fiber mats and serving to return recovered stock is different. In fact the auxiliary equipment is unique in many ways and particularly so because it is completely automatic.

The full scale plant is designed to handle 5600 g.p.m. of waste water having a fiber concentration of 1.6 pounds per thousand gallons. At the design flow rate the cylinder speed of the Waco filters is 40 f.p.m., and the effluent water fiber concentration can be less than 0.1 pound per thousand gallons with sufficient mat stock on the wires. With more or less fiber in the incoming waste water the hydraulic capacity is affected as shown before in Fig. 3.

The simplified flow diagram (Fig. 8) shows the general operation. Waste water from the collector tank is piped into the inlet box and distributed evenly across the width of each filter. Precoat mat stock is brought to the plant at 3% consistency and metered as to volume through a stock meter in accordance with the prevailing flow rate to make a constant mat weight. It is then diluted to 0.5% and pumped to the precoat vats of each filter. Recovered and mat stock are pumped to an agitated tile chest from each filter and regulated to 3% consistency before being returned to the stock preparation area of the mill. Effluent water from each filter passes through a weir box for volume measurement, then to the sewer, and finally to the Delaware River. An automatic sampler determines the average fiber content of the filtered water.

It is desirable to examine the auxiliary equipment more closely to see how the automatic controls are used. A level recorder controller accelerates or decel-

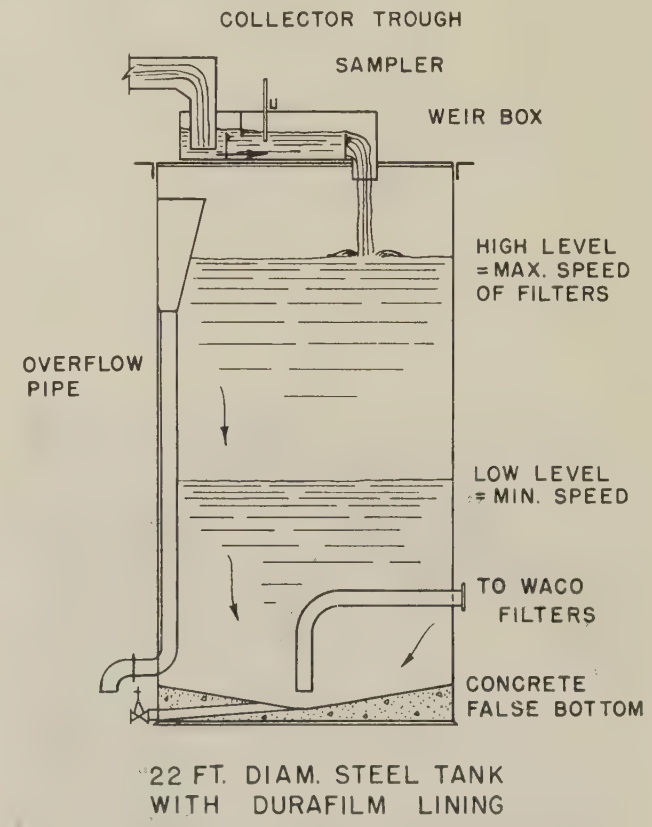

Fig. 6. Collector tank

erates the Waco filters in accordance with the level in the collector tank. This instrument is the watchdog of the plant. It also regulates the speed of the stock meter to provide just enough mat stock to satisfy the prevailing filter speed. Since the collector tank is large

in size it is possible to operate the controller for a wide range of sensitivity to avoid sudden increases or decreases in filter speed. The controller gradually changes the filter speed when the collector tank level increases. The adjustment is made to have the filter speed at a maximum just below the overflow level in the collector tank.

The stock meter speed is related to the collector tank level as noted above. It is also possible to adjust the stock meter speed to compensate for the number of Waco filters in operation and to obtain any desired mat weight at a given speed. This flexibility is obtained by a manual setting of a variable speed drive and a rheostat controlling the field of the direct-current motor.

Level controllers are used to regulate the levels of stock in the mat stock chest and the recovered stock chest. If the stock pump on the recovered stock chest should happen to shut down, an electrical-pneumatic interlock closes the control valve and thus acts like a check valve.

The incoming waste water flows over a weir designed to handle 7000 g.p.m. and equipped with a level recorder. Since the weir formula shows that quantity varies as the three-halves power of head over the weir, it was desirable to provide a level recorder having a chart with a three-halves power spacing to enable the operator to read flow in gallons per minute directly and to be able to integrate the total flow.

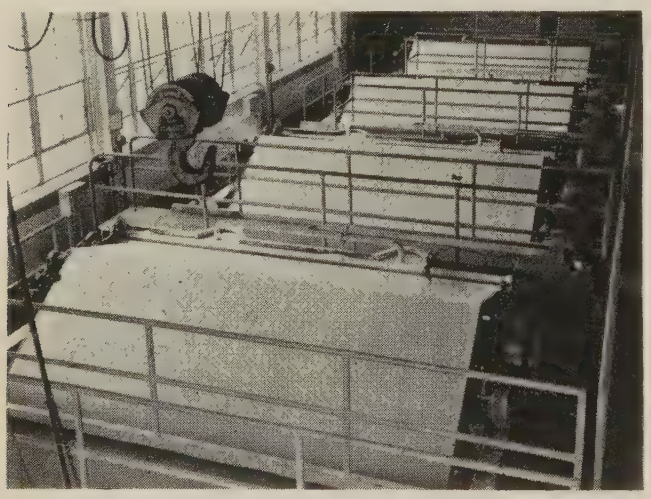

Fig. 7. Interior view of waste water disposal plant

As might be expected level instruments were employed for the control of level in each of the precoat vats and the main filter vats. A level controller was also used on the dilute mat stock chest.

The automatic control of recovered stock at each filter posed an unusual problem because it was desired to pump the recovered stock to the storage chest at intermittent intervals and the consistency had to be regulated. There is insufficient stock to make a continuous pump-out arrangement feasible so the individual pumps are used to circulate stock in the boxes until enough stock is accumulated. When the level in the boxes reaches a set height a control operates a three-way valve to transfer recovered stock to storage. Using the pumps for circulation has the advantage of mixing dilution water with the recovered stock to obtain a uniform consistency. The quantity of dilution water is determined by a flow controller which is reset by a speed transmitter mechanically driven by the stock meter.

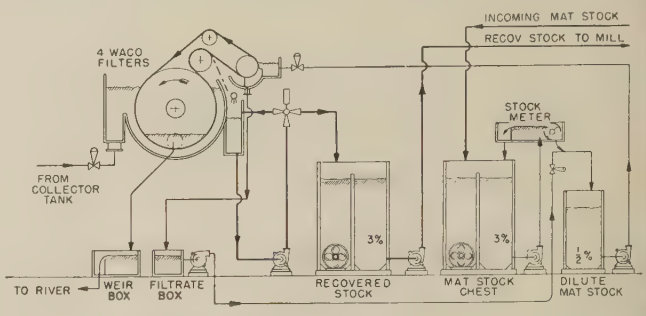

Fig. 8. Simplified flow diagram of waste water disposal plant

Table II. Waste Disposal Plant Operation

Month	Consistency, lb./1000 gal. Inf.	Consistency, lb./1000 gal. Eff.	Reduction in consistency, %	B.O.D., p.p.m. Inf.	B.O.D., p.p.m. Eff.	B.O.D., p.p.m. Reduction, %	Settleable solids, % reduction	Operation, hr./day
April	1.03	0.10	90.3	222	116	46.6	No data	23.8
May	0.88	0.10	88.6	308	162	46.9	87.9	24.0
June	0.79	0.12	84.9	237	159	32.7	96.5	23.8
July	0.90	0.18	80.1	219	155	29.5	98.3	23.9
Aug.	0.90	0.16	82.2	212	167	19.7	97.8	23.9
Sept.	0.91	0.16	82.4	197	113	42.6	93.8	23.8
	0.90	0.14	84.8	233	145	36.3	94.9	23.9

The speed transmitter provides a pneumatic impulse in a ratio equal to the square root of the speed to reset the flow controller. It will be recalled that the speed of the stock meter is proportional to the flow of mat stock and since this flow represents 80% of the total fiber it is possible to control consistency very accurately.

PLANT OPERATION

Up to now, pilot plant studies and design and construction of the full size plant have been described.

With any project of this kind it is always of interest to determine how close the completed project came to predictions. Since this plant was placed in operation daily records of its performance have been maintained not only to establish its performance, but also to provide data for periodic reports to the Commonwealth of Pennsylvania for their use.

The essence of the last report submitted to the Commonwealth is shown on Table II. It is a summary of the initial six months' operation.

Several observations may be made from this tabulation. The first and most satisfactory is the fact that the filters have lived up to expectations by performing

most creditably. This is attested to by the record of almost continuous operation during the initial 6 months. Capacity of each filter has consistently been in the 800 to 1200 g.p.m. range depending of course upon the characteristics of the influent and as can be seen from the tabulation, the quality of the effluent has averaged 0.14 pound per thousand gallons for the same period. The per cent reduction in consistency has not been quite up to prediction caused principally by the lower fiber content of the influent than was originally estimated. It is safe to say and occasional operating conditions have shown that the quality of the effluent remains substantially unchanged with greater fiber content in the influent. Thus had the influent averaged 1.5 pounds per thousand gallons, the reduction in consistency would have exceeded 90%.

B.O.D. reduction has come very close to predictions made from pilot plant studies. The tabulation shows that the reduction in settleable solids has been excellent: a maximum of 98.3% having been achieved during the month of July.

Mechanical operation of the filters, controls, and auxiliaries has been excellent. This being the first plant of its type ever designed and built in this country, it would not have been surprising had a number of changes been required. Actually little difficulty was experienced during start-up, only adjustments to some control valves and controllers were found necessary. No difficulty has been experienced with freezing in the open collector trough or tank, the water velocity being sufficient to prevent freezing.

Initially, little was known of the life that could be expected from the traveling wires. Experience with the pilot unit in this respect was most inconclusive for a number of reasons. Generally, however, life had been relatively short. Improvements incorporated in the filter design and better operating conditions combined to extend wire life to about 6 months. Further improvement in wire life can be expected from the development of stainless steel or Monel wires for this application.

It is of interest to observe some of the effects of recovered fiber on the mat stock. Freeness was reduced about 200 points, brightness about 3.5 points, and fractionation about 8 points. These changes are so insignificant that it is not possible to detect any alteration in quality to the finished paper.

The principal reason for the plant's existence is its contribution to the over-all clean streams program. Of almost equal importance is its economic effect on company operations and in a plant of this type, it may be said that if the recovered fiber can be used without having a deleterious effect on the finished paper then the plant may at least be partially self supporting. For this particular plant, the recovered fiber amounting to about $1^1/_2$ tons per day contributes something toward operational costs but probably is not nearly sufficient to make the plant self supporting. Beyond this consideration is the effect of general pollution abatement on the stream and municipalities using it for recreational and drinking purposes. In the past, it has been common practice to install extensive water purification works upstream from a city and to discharge raw sewage downstream. Extensive installation of waste treatment plants by both industry and municipalities will materially improve the streams and rivers and make extensive filtration plants unnecessary. Needless to say, the new treatment plant at the Scott Paper Co. has contributed to the over-all stream improvement program.

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Polyamide Resins for Paper Coating

HAROLD WITTCOFF, M. M. RENFREW, and FRED B. SPEYER

Polyamide resins, condensation polymers of dimerized and trimerized vegetable oil acids with diamines, are suited for paper coatings because they make the paper functional by imparting to it heat sealing properties as well as resistance to water vapor, grease, solvents, and chemicals. Three basic types of resins are available, and all three may be coated as hot melts, as solvent solutions, and as water dispersions. This paper discusses the compatibility relationships of both the solid resin and the suspensoid form. The uses for the coatings, and coating techniques are reviewed.

Polyamide resins comprise a group of polymeric materials which derive from the condensation of dibasic organic acids with diamines or from the self-condensation of certain ω-amino acids. Products with widely differing properties are obtained by varying the selection of acids and amines, but in general they are characterized by sharp melting points unless chemically modified or compounded with other materials.

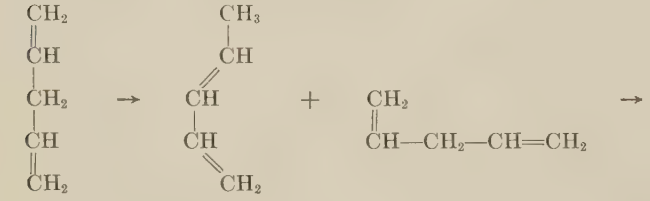
Perhaps the best known of the polyamides is the condensation product of adipic acid with hexamethylene diamine. This nylon, hexamethylene adipamide, has many properties which would be desirable in a paper-coating material if it could be applied conveniently. However, it is low in solubility; it is extremely high softening (above 500°F), and it tends to char rapidly at the melting point unless protected from contact with air. Such properties prevent the application of hexamethylene adipamide to paper by the method normally available to the converter.

For coating on paper there have been developed polyamide resins which are lower softening and more readily handled. The work, which is now reported, has been restricted to a special group of these lower softening polyamide resins formed by the condensation of dimerized and trimerized vegetable oil acids and diamines

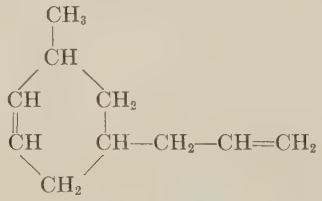
Harold Wittcoff, M. M. Renfrew, and Fred B. Speyer, Paper No. 124, Journal Series, Research Laboratories, General Mills, Inc., Minneapolis, Minn.

or triamines. These materials, known as the General Mills Polyamide Resins, within themselves, cover a wide range of properties. The physical forms in which they are available for coating, their properties in thin films, their uses in paper converting, methods of application, and their compatibility relationships with other resins and plasticizers will be discussed.

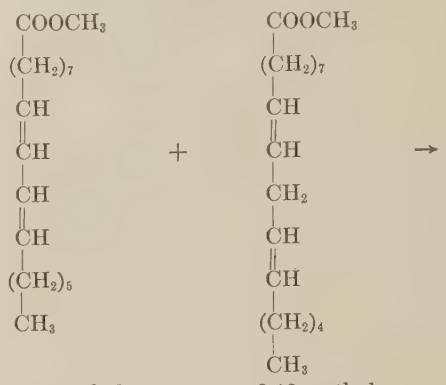
The polyamide resins, as indicated above, are the condensation products of di- or triamines with dimerized and more highly polymerized vegetable oil acids. They were initially described by Cowan, Falkenberg, Teeter, and Skell (*1*). The component of vegetable oils which is most prone to dimerize and produce the dibasic acid required for condensation with the diamine is 9,12-linoleic acid. That the dimerization proceeds via a so-called Diels-Alder mechanism may be assumed from the behavior of 1,4-pentadiene, a simple analog of 9.12-linoleic acid. This unconjugated diene was shown by Ahmad and Farmer (*2*) to yield a dimer, under the influence of heat, which could result from the Diels-Alder condensation of conjugated 1,3-pentadiene with unconjugated 1,4-pentadiene. Thus heat could have caused a portion of the di-unsaturate to seek its more stable or conjugated form which in turn could condense with the unchanged material to yield a dimer.



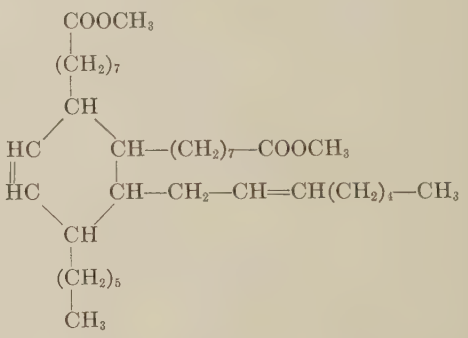
1,4-pentadiene 1,3-pentadiene 1,4-pentadiene



Analogously to the simpler situation, it seems logical to postulate that 9,12-linoleic acid dimerizes in a similar manner. In 1941, Bradley and Johnston (*3*) isolated by fractional molecular distillation a dimer fraction from the heat-induced polymerization of methyl linoleate from dehydrated castor oil. It may be assumed that a portion of the methyl linoleate isomerized to provide a conjugated diene which underwent a Diels-Alder reaction with one of the double bonds of either the unconjugated or the conjugated linoleic acid. As a point of departure, the following reaction may be postulated.



9,11-methyl linoleate (isomerized) 9,12-methyl linoleate



Obviously, the possibilities for isomerism are manifold. The 9,12-double bond structure may conjugate either to the 9,11 structure, as shown above, or to a 10,12 structure. Either of these conjugated dienes may condense with either of the double bonds of the 9,12, the 9,11, or the 10,12 compounds. Finally, these condensations may occur either "head to head" or "head to tail." More definitive work on the chemistry of this interesting material has been carried out by Wheeler (*4*), Paschke, Wheeler, and Jackson (*5*), and Paschke and Wheeler (*6*). These papers may be consulted for more elaborate discussion of the chemistry involved. In any case, however, the dibasic acid which forms the basis for General Mills Polyamide Resins may be assumed to have the structure indicated in the above formula.

Table I. Specifications of Polyamide Resins

	Polyamide Resin		
	No. 93	*No. 94*	*No. 95*
Viscosity at 25°C.[a]			
Cp.	65–100	50–85	14–50
Gardner-Holdt	B-D	A-C	A_1-A
Color (Gardner)[a]	12 (max.)	12 (max.)	12 (max.)
Acid number	7 approx.	7 approx.	7 approx.
Ash, % by weight	0.10 (max.)	0.10 (max.)	0.10 (max.)
Specific gravity	0.97–0.99	0.97–0.99	0.96–0.98
Pounds per gallon	8.1–8.3	8.1–8.3	8.0–8.2
Penetration at 25°C.[b]	5 approx.	7 approx.	20 approx.
Ball and ring softening point[c]	105–115°C.	105–115°C.	95–105°C.
Average molecular weight	6000–9000	3000–6500	3000–6500

[a] Determined on 35% solution in toluene, *n*-butanol (1 : 1).
[b] ASTM standard method with 200-gram weight.
[c] ASTM standard method.

PROPERTIES OF POLYAMIDE RESINS

Three basic types of polyamide resins, based on polymerized vegetable oil acids, are available. The first of these is the condensation product of ethylene diamine and dimerized and trimerized vegetable oil acids. This resin is a hard, amber-colored substance of medium melting point and, as will be seen below, of rather limited solubility. It is nonblocking and exhibits very little cold flow or deformation under stress. This ethylene diamine-containing resin is manufactured in three different forms known as Polyamide Resins Nos. 93, 94, and 95, respectively. The nos. 93 and 94 resins are similar save that the former has a molecular weight range of 6000 to 9000 as compared to 3000 to 6500 for the latter. The no. 95 resin comprises a mixture of the no. 94 resin with small amounts of modifiers which in-

clude paraffin, the glycerol ester of hydrogenated rosin, and N-ethyl *o*- and *p*-toluenesulphonamides. The specifications of these three resins are indicated in Table I.

The second basic type of resin, known as Polyamide Resin No. 100, is the condensation product of diethylene triamine and dimerized and trimerized vegetable oil acids. Unlike its ethylene diamine counterpart, this resin is a tacky, low melting, plasticizing resin with a marked tendency to exhibit cold flow. It is generally lighter in color than the ethylene diamine resin and, as will be seen later, has a wider range of solubility and compatibility. Its properties are indicated in Table II.

The final type of resin, termed Polyamide Resin No. 90, is a copolymer prepared by condensing ethylene diamine with dimerized and trimerized vegetable oil and other acids. The no. 90 resin is considerably higher melting, harder, and less soluble than the no. 93 resin. It exhibits virtually no cold flow and is highly resistant to blocking. Its properties are described in Table III.

All of these resins are available in stabilized modifications which are described below.

PHYSICAL FORMS OF POLYAMIDE RESINS

Polyamide resins are available for use as hot melts, as solvent solutions, or as water dispersions which are termed suspensoids. These three methods for application to surfaces make the resin a very versatile coating material for the paper converter.

Hot Melt Resins

All of the polyamide resins, either alone or in combination, can be coated as hot melts. The no. 94 resin is particularly valuable here because its melting point is lower than that of most thermoplastic resins, and the correspondingly low temperature of manipulation does not endanger the strength of the paper.

The no. 95 resin alone is an excellent hot melt composition with improved water vapor resistance because of the paraffin present. The no. 90 resin is seldom used alone as a hot melt coating because of its high melting point and brittle nature. When properly plasticized and compounded with the other polyamide resins, however, it works well.

Actually, both the nos. 94 and 90 resins may be used in combination with the no. 100 resin which plasticizes and flexibilizes them and increases their sealing range after heat activation. Furthermore, compositions containing the no. 100 resin seal well to nonporous surfaces such as cellulose acetate. The high blocking resistance of the nos. 94 and 90 resins makes it possible to formulate nonblocking compositions containing large quantities of the no. 100 resin. As a matter of fact, compositions containing 50% polyamide resin no. 100, 30% of the no. 90 resin, and 20% of the nos. 94 and 95 resin are nonblocking under severe conditions.

Table II. Specifications of Polyamide Resin No. 100

Viscosity at 25°C.[a]	
Cp.	85 (max.)
Gardner-Holdt	C (max.)
Color (Gardner)[a]	12 (max.)
Acid number	12 (max.)
Ash, % by weight	0.10 (max.)
Specific gravity	0.99
Pounds per gallon	8.24
Ball and ring softening point[b]	43°C. (min.)
Average molecular weight	3000–6500

[a] Determined on 35% solution in toluene, *n*-butanol (1:1).
[b] ASTM standard method.

Table III. Specifications of Polyamide Resin No. 90

Viscosity at 25°C.[a]	
Cp.	50–100
Gardner-Holdt	A-D
Color (Gardner)[b]	12 (max.)
Acid number	15 (max.)
Ash, % by weight	0.10 (max.)
Specific gravity	0.98
Pounds per gallon	8.1
Penetration at 25°C.[c]	2(approx.)
Ball and ring softening point[d]	173 (min.)

[a] Determined on 25% solution in phenol, *n*-butanol (1:1).
[b] Determined on warm 35% solution in toluene, *n*-butanol (1:1)
[c] ASTM standard method with 200-gram weight.
[d] ASTM standard method.

The behavior of the polyamide resins in coating machines is generally considered excellent. Because of residual unsaturation in the dimerized fatty acids, however, as well as because of the possibility of the presence of free amine end groups, a tendency exists for the formation of "skin" or oxidized particles when the resin is exposed to air at high temperatures for long periods of time, or when it is repeatedly remelted after solidification. To overcome this, stabilized forms of the various resins have been developed. These stabilized resins possess cure times at 200°C. of at least 1800 seconds, and are designated by the addition of the letter S to the regular code number.

Several antioxidants have been found to stabilize the resins and these include *p*-phenylphenol, di-*tert*-butyl-*p*-cresol, butylhydroxyanisole, Ionol, Flectol, Santoflex BX, di-"lauryl"-thiodipropionate and thiodipropionic acid. In general, concentrations of 0.01 to 1.0% are effective.

Hot melt coating of Polyamide Resins Nos. 93, 94, or 95 is ordinarily accomplished at 140 to 180°C., for it is within this range that a definite "break" in melt viscosity occurs. The relationship of melt viscosity to temperature, as determined with a MacMichael viscosimeter, is demonstrated in Fig. 1.

The hot melts based on ternary mixtures of the nos. 90, 100, and 94 resins must be coated at higher temperatures, which, however, are still within the limits set by paper converters.

Resin Solutions

As indicated previously the no. 100 resin demonstrates the greatest degree of solubility and the no. 90 resin demonstrates the least. The nos. 93, 94, and 95 resins are intermediate in their solubilities. Although solubility of the resins is limited, the limitations are actually advantageous in practice. This follows since the most important practical primary solvents for the nos. 93, 94, and 95 resins are alcohols higher than methyl alcohol, primarily isopropyl and butyl alcohols. Alcohols are less flammable than the solvents which must be used with most resins. Also this limited solubility, as will be seen later, may be correlated directly with the excellent solvent resistance of the resin coatings.

Other primary solvents of a less practical nature include cyclohexanol, methyl isobutylcarbinol, 2-nitro-1-butanol, chloroform, methylene chloride, trichloroethylene, chlorobenzene, and acetic acid.

Secondary solvents include benzene, toluene, xylene,

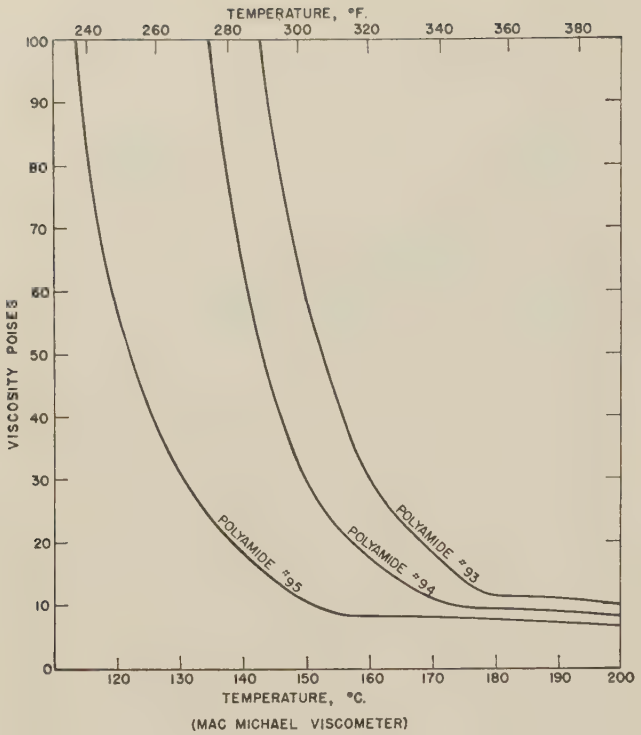


Fig. 1. Viscosity (poises) of melted polyamide resins vs. temperature

mineral spirits, naphtha, cellosolve, butyl cellosolve, carbitol, methyl carbitol, methyl cellosolve, amyl acetate, butyl acetate, methyl ethyl ketone, and nitrobenzene. Many of these secondary solvents become primary solvents when hot. If necessary, considerable amounts of almost any common solvent can be used as a diluent, provided at least 30% of a primary solvent is present.

The solvency of the resins is greatly improved by the addition of the secondary solvents to the primary ones. Thus, mixtures of isopropyl alcohol with hydrocarbons in the ratio of 30:70 to 50:50 are the most common solvent combinations. With these solvents, solutions with concentrations up to 40% can readily be made. The alcohol-hydrocarbon combinations may be modified by the addition of methyl ethyl ketone or ester solvents such as butyl acetate, especially when solutions including nitrocellulose are desired. A typical combination is isopropyl alcohol, methyl ethyl ketone, and hydrocarbon in the ratio of 20:20:60. For extremely rapid evaporation a solvent combination of ethanol, acetone, and hexane has been used.

In general, Polyamide Resin No. 100 is more soluble in all of the above-mentioned solvents than are the nos. 93, 94, and 95 resins. Thus, the no. 100 resin is soluble in hot ethanol in all proportions, whereas cold ethanol is soluble in the resin in concentrations up to 50%.

Complete solubility of the no. 90 resin is attained in solvents such as phenol or dimethylformamide, or combinations of these with alcohols. Solutions with concentrations as high as 35% can be made in alcohols higher than ethanol, or better, in combinations of alcohols and hydrocarbons, provided that these solutions are kept warm. On cooling, immobilization or gel formation occurs at high solids contents. At low solids concentrations, the solutions are thixotropic, and their high viscosity can be reduced by agitation.

Resin Suspensoids

Polyamide resin suspensoids are cationic, water dispersions of the resins described above. They are described in some detail by Wittcoff (*7*) and by Wittcoff, Peerman, Speyer, and Renfrew (*8*). The procedure of dispersion is such that the films resulting from the suspensoid are comparable in every way to those obtained from the solvent or hot melt application. Five modifications are now produced which are based on the nos. 94 and 95 resins and which differ from each other in that some have been compounded further after dispersion. Suspensoids based on the nos. 90 and 100 resins are under development. These products vary in solids contents from 35 to 50% and in viscosity from 0.4 to 15 poises. Their mechanical stability is good since many of the compositions can withstand repeated freezing and thawing or extensive agitation without injury. The average particle size of these suspensoids is 1 mμ, and their pH varies from 4.0 to 5.5.

The suspensoids are coated readily to provide opaque films composed of minute particles which are practically indistinguishable, unless examined under magnification. Because these films are discontinuous, it is imperative that they be fused if their maximum value as coatings is to be achieved. Obviously, fusion is not necessary if heat sealing is the only property desired. The fusion temperature of most of the suspensoid films is slightly above the boiling point of water. Fusion may also be obtained by the incorporation of solvents or plasticizers. The wet films on the web are usually passed through ovens where the drying and fusion operations are accomplished in one step. Since only water is involved, gas-fired ovens are satisfactory. The temperature of the oven should not exceed 350°F. since too rapid evaporation of the water leads to the formation of pitted surfaces. The residence time in the oven is a function of the thickness and porosity of the paper used, the thickness of the coating, and the temperature of the oven. It may vary from 15 to 90 seconds.

By way of comparison, polyamide resin suspensoid films dry to constant weight in 9 to 14 minutes at 50% relative humidity and 73°F. A solvent solution (35% in isopropyl alcohol-toluene, 1:1) under the same conditions reaches constant weight in eight minutes.

PROPERTIES OF POLYAMIDE RESIN FILMS

Most papers offer little more than a point of departure for the preparation of a barrier. Once the paper has been manufactured, it must be made functional by the application of a coating. Polyamide resin films, whether applied from hot melt, solvent, or suspensoid

Table IV. Moisture Vapor Transmission of Polyamide Resin Films

Composition used	*Water vapor permeability, g./m.²/24 hr.* *Flat*	*Fold*
Control	181	...
Polyamide Resin No. 94, from solvent	8.3	9.5
Polyamide Resin No. 95, from solvent	0.5	0.7
Polyamide Resin No. 95, from suspensoid (fused)	2.3	3.5
Polyamide Resin No. 90, from solvent	20.8	21.0
Polyamide Resin No. 90: Resinous Alcohol (80:20), from solvent	18.2	17.6
Polyamide Resin No. 90 with 4% paraffin, from solvent	0.8	1.8

Table V. Greaseproofness of Polyamide Resin Films

Composition used	Time for turpentine penetration sec. — Flat — No. of tests	Min.	Max.	Av.	Fold — No. of tests	Min.	Max.	Av.
Polyamide Resin No. 94, from solvent	2	1800+	1800+	1800+	4	1800+	1800+	1800+
Polyamide Resin No. 95, from solvent	2	1800+	1800+	1800+	4	1800+	1800+	1800+
Polyamide Resin No. 95, from suspensoid (fused)	2	1800+	1800+	1800+	10	60	1800+	1245+
Polyamide Resin No. 90, from solvent	2	1800+	1800+	1800+	10	625	1800+	1140+
Polyamide Resin No. 90: Polyamide Resin No. 100, (40:60), from solvent	2	1800+	1800+	1800+	4	1800+	1800+	1800+
Polyamide Resin Suspensoid B-1001X (fused)	2	1800+	1800+	1800+	10	30	1800+	1621+

serve to make paper resistant to the transmission of moisture vapor. At the same time, unlike most water vapor barriers, they provide greaseproofness. Solvent and chemical resistance are also imparted to the paper, as are excellent heat sealing properties, and marked resistance to blocking. Each of these properties will be discussed in turn. Aging stability of the coatings appears to be excellent, as indicated by both accelerated aging and long-term tests. Coated papers, stored under laboratory conditions for 5 years, have demonstrated no functional deterioration. It is possible to print on the resin coatings, although some difficulty may be encountered with the no. 95 resin coating since paraffin tends to accumulate at the surface.

Moisture Vapor Transmission

Moisture vapor transmission tests were carried out at 72°F. and 65% relative humidity, according to tentative TAPPI Standard T 448 m-40. All of the compositions were coated on 23-pound cereal glassine at 8 pounds per ream (20 × 25—500). Transmission was determined on both flat surfaces and on folds. The results are indicated in Table IV where it is apparent that the no. 94 resin itself provides a barrier which, because of the flexibility of the resin, is as effective on a fold as on a flat surface. This holds true for all of the resins tested, and is of great importance in packaging applications. Almost complete water vapor resistance is demonstrated by films of the no. 95 resin cast from solvent, due no doubt to the presence of a small quantity of paraffin. It is significant that films from this same resin when cast from aqueous suspensoid still demonstrate excellent water vapor resistance. The retention of this important property by the suspensoid is possible because of the unique procedure employed for dispersion. The no. 90 resin has less water vapor resistance, but it is significant that it can be plasticized with Resinous Alcohol (General Mills, Inc.) without further harming the water resistance. The addition of a small quantity of paraffin to the no. 90 resin improves markedly its water vapor resistance.

Table VI. Chemical Resistance of Polyamide Resin No. 94 and Vinyl Chloride Copolymer Coatings

Chemical	Time of immersion, hr.	Condition of film — Polyamide No. 94	Vinyl chloride copolymer
Hydrochloric acid (1 *N*)	24	o.k.	o.k.
Acetic acid (20%)	24	o.k.	o.k.
Ammonium hydroxide (10%)	24	sl. blush	blush
Sodium hydroxide (10%)	24	sl. blush	blush
Sodium chloride (20%)	24	o.k.	o.k.
Mineral oil	24	o.k.	o.k.
Soybean oil	24	o.k.	o.k.
Motor oil	24	o.k.	o.k.
Methyl ethyl ketone	24	sl. blush	dissolves
Ethyl alcohol (95%)	24	o.k.	blush
Insecticide ("Bug-a-boo")	24	blush	o.k.
Cigarette lighter fluid	24	o.k.	o.k.

Greaseproofness

Greaseproof tests were carried out according to TAPPI Standard T 454 m-44 with turpentine. For fold tests the paper was creased and the crease was subjected to the action of a 500-gram weight. All of the compositions were coated on 23-pound cereal glassine at 8 pounds per ream (20 × 25—500). The results are indicated in Table V. It is obvious that the nos. 94 and 95 resins and a mixture of the nos. 90 and 100 resins have excellent grease resistance.

Chemical and Solvent Resistance

Polyamide resin films demonstrate excellent resistance to alkalis, dilute acids,* oils, solvents, and proprietary products containing solvents. In order to compare the solvent resistance of Polyamide Resin No. 94 films with films of a vinyl chloride copolymer commonly used by paper converters, tin strips were dipped in solutions of both resins and were allowed to drain. The Polyamide Resin No. 94 solution was made with toluene-isopropyl alcohol (1:1), whereas the vinyl chloride copolymer solution was made with methyl ethyl ketone-toluene (1:1). After the strips had drained, the polyamide coatings were heated at 120°C for 10 minutes whereas the vinyl coating was heated at 135°C. for 15 minutes. Temperatures above 135°C. are not practical in most paper converting operations. The strips were then immersed in various chemicals for 24 hours. The results are indicated in Table VI.

Hot Sealing Properties

One of the outstanding properties of polyamide resin films from the nos. 93, 94, and 95 resins is the ability of these films to provide heat seals at the low temperature of 80°C. and, at the same time, to demonstrate excellent blocking resistance. The low sealing temperature makes possible rapid machine operation and insures lack of damage to the paper. These films provide bonds for a wide variety of surfaces including paper label stock, kraft paper, glassine paper, waxed papers, waxed glassine, metal foils, metals, glass, cloth, cork, asbestos, leather, wood, and coated cellophanes.

The no. 90 resin is not used alone as a heat sealing material because of its brittleness. When mixed with the nos. 93, 94, or 95 resins, however, it adds strength to the bond and tends to prevent the resin in the molten state from penetrating porous surfaces. The no. 100 resin, likewise, is not used alone as a heat seal coating

* Polyamide Resin No. 100 is not acid resistant because of its high content of free secondary amine groups.

because of its tackiness, which, however, fits it for a pressure-sensitive type of laminating adhesive. The other resins serve to detackify the no. 100 resin; and in admixture with them, the no. 100 resin imparts to the resultant composition the ability to seal to nonporous surfaces such as those exhibited by PT cellophane and cellulose acetate.

Although the sealing temperature of the nos. 93, 94, and 95 resins is as low as 80°C., the sealing range does not extend beyond 100°C. An elongated sealing range is important since sealing temperatures are poorly controlled in many machines. What is perhaps more important is the fact that an elongated sealing range is an indication that a seal will withstand tension while still warm. Extension of the sealing range has been accomplished by blending the no. 100 resin with the nos. 90 or 94 resins, or better by making ternary mixtures of the nos. 100, 90, and 94 resins in the proportion of 50:30:20. Furthermore, suspensoid compositions are available which have been formulated to seal as low as 70°C. and which have sealing ranges extending beyond 150°C. At the same time these suspensoid compositions demonstrate excellent blocking resistance.

Antiblocking Properties

The resistance of polyamide resin films to blocking has been measured by TAPPI Standard T 477 m-47. The maximum blocking conditions which can be withstood by various polyamide resins is indicated in Table VII. These tests were carried out face to back with coatings on label paper at 4 pounds per ream (20 × 25—500). Temperatures beyond 60°C., time beyond 7 days, and pressure beyond 10 pounds were not investigated since conditions more severe than these are practically never encountered in actual practice.

USES OF POLYAMIDE RESIN COATINGS

Polyamide resin coatings may be used as water vapor resistant, greaseproof, oil and chemical resistant coatings which in addition have good heat sealing properties and marked blocking resistance. More specifically, they find use as coatings for kraft paper and cellophane, for certain plastic films, and for paperboard where greaseproofness is desired.

As a gloss overcoat for labels, paperboard cartons, printed cellophane, and other packaging materials, the resin provides a grease and moisture vapor barrier together with a degree of structural strength. In addition, it provides gloss in such an application, and its ability to be pigmented offers interesting possibilities. Still another advantage in this application arises from the nonflammability of the resin.

One of the biggest uses of the resin is in the field of heat seal coatings. Its properties in this regard have already been discussed above. It is an excellent adhesive for bonding labels to various surfaces. It is used in making bag seals and in numerous other applications requiring the formation of bonds under the influence of heat.

Because of its adhesive properties the resin may be used for the preparation of laminated structures. Its versatility makes possible the bonding together of many combinations of dissimilar surfaces so that advantage may be taken, in the final composition, of the properties of both. Thus, in addition to serving as an adhesive

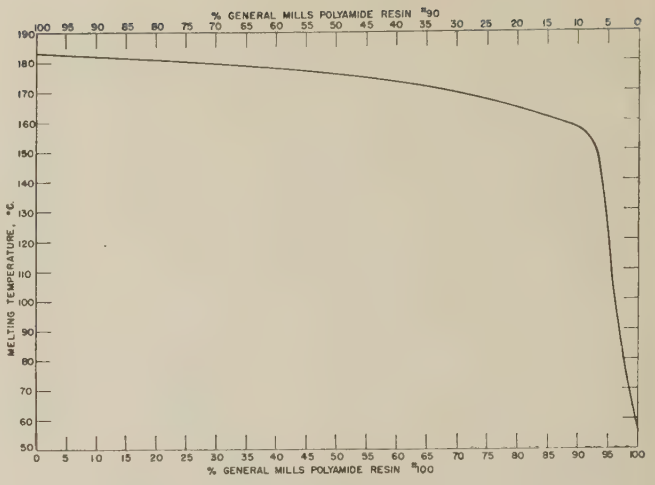


Fig. 2. Softening points of mixed Polyamide Resins Nos. 90 and 100

for the lamination of various types of paper such as kraft, sulphite, and glassine, it serves to laminate substances such as foil, cellophane, cork, rubber, metal, glass cloth, and asbestos to themselves and to each other. Polyamide resin suspensoids may be combined with wax emulsions to improve markedly the adhesion of the latter.

Polyamide resin in the suspensoid form is, or can be made, compatible with a large number of widely used paper coatings such as starch, dextrines, clays, and gums. It imparts much needed water resistance to these even when used in small quantities. The resin suspensoids may be used as components of tub size compositions, and, in addition, compositions are available which may be added to paper during its fabrication in order to improve wet-strength properties.

Miscellaneous uses include the application of the resin as a resistant coating for paper bottle cap liners, as a coating for fiber oil containers, as a coating for improving the transparency of window glassine, as a nonblock backing for adhesive or pressure-sensitive tapes, as a

Table VII. Blocking Properties of Polyamide Resin Films

Composition	*Max. temp., °C.*	*Max. relative humidity, %*	*Max. pressure, p.s.i.*	*Max. time, days*
Polyamide Resin No. 94, from solvent or hot melt	60	100	10	7
Polyamide Resin No. 95, from solvent or hot melt	60	100	10	7
Polyamide Resin No. 90, from solvent or hot melt	60	100	10	7
Polyamide Resin Suspensoid A-001x[a]	60	75	1	5–7
Polyamide Resin Suspensoid A-000[b]	60	75	1	7
Polyamide Resin Suspensoid B-200[c]	60	75	1	7
Polyamide Resin Suspensoid B-1001x[d]	60	75	1	7
Polyamide Resin Suspensoid E-200[e]	60	75	1	5–7
Polyamide Resin Suspensoid A-003[f]	60	75	1	5–7
Polyamide Resin Suspensoid A-004[g]	60	100	1	7

[a] Based on no. 94 resin.
[b] Based on no. 95 resin.
[c] Based on no. 95 resin and compounded.
[d] Based on no. 94 resin and compounded.
[e] Based on no. 95 resin and compounded.
[f] Experimental composition based on no. 90 resin.
[g] Experimental composition based on no. 90 resin.

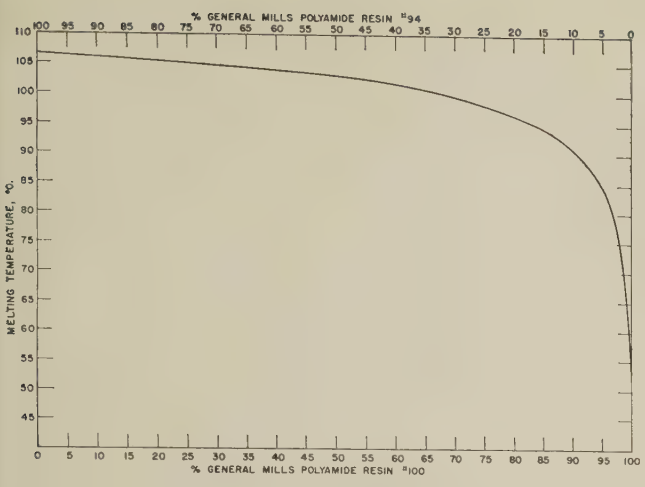


Fig. 3. Softening points of mixed Polyamide Resins Nos. 94 and 100

wallpaper sealer, and as a binder for pigment coatings for paper.

Solvent solutions of the resin as well as the suspensoid may be pigmented or colored with dyes. This represents intriguing possibilities to the ink formulator. Also, the ability of the resin to adhere to many surfaces makes it a likely prospect for use as an "anchor" coating for cellophane and plastic films.

COMPATIBILITY RELATIONSHIPS

Compatibility of Polyamide Resin with Other Resins and Plasticizers

In order to arrive at the optimum compositions for the uses described above, it is frequently necessary to compound the resins. The compatibility relationships of polyamide resin and polyamide resin suspensoids have been widely studied and are detailed in technical bulletins on these products issued by General Mills, Inc. (*9*, *10*). Polyamide resin, in general, is entirely compatible with rosin and rosin derivatives, which tend to tackify it and lower the cost of the final composition. It is compatible with most phenolic and maleic resins. Paraffin and microcrystalline waxes can be added to the resin to the extent of about 4%. Other waxes, including chlorinated waxes, demonstrate a greater degree of compatibility. Waxes, in general, improve the water vapor resistance of the resin and make it even more nonblocking. Larger quantities of waxes can be incorporated into the resin if almost equally large quantities of rosin or rosin esters are present to serve as fluxes. Kenflex A (Kenrich Corp.), a methylnaphthalene-formaldehyde resin, acts as a flux as do various fatty acid amides. The use of amides as fluxes is described in a bulletin issued by Armour and Co. (*11*). The resin is, in general, incompatible with most vinyl, alkyd, urea, melamine, and petroleum resins. This latter statement is not to be taken as a hard and fast generalization, however, since complete compatibility is demonstrated with the styrene resin, Piccolastic A-25 (PICCO) which is an excellent extender. Polyamide resin demonstrates some compatibility with the polybutenes (Oronite) and with low molecular weight polyisobutylene (Vistanex, Enjay Corp.) which tackifies it and improves its cold temperature properties. It is compatible to some degree with polyethylene (Bakelite DYGT), which likewise improves the low temperature properties of the resin. In borderline situations, compatibility may be demonstrated in the melt form but not in solution. With SS nitrocellulose (Hercules), however, it is compatible in all proportions in solution, whereas with RS nitrocellulose, it is compatible if the proportion of resin to nitrocellulose is less than 4 to 1. Nitrocellulose serves to make the resin even more nonblocking.

An acrylic resin, Acryloid A-10 (Rohm and Haas) is compatible in small concentration. Some coumarone-indene resins, such as Nevillac soft and Nevillac hard (Neville Co.) demonstrate compatibility as do Nevillac OP, RA, RP, RS, and RT which are phenol-hydrocarbon resins. The sulphonamide resins known as Santolites (Monsanto) are for the most part compatible and serve as tackifiers.

Polyamide resin is compatible with many liquid and low melting monomeric plasticizers, among the best of which are Santicizer 8, which is N-ethyl *o* and *p*-toluenesulphonamide (Monsanto), and tributyl phosphate. Resinous Alcohol (General Mills, Inc.) a partial glycerol ester of mixed rosin and fatty acids, is an excellent plasticizer. Of particular value as a plasticizer for the nos. 90, 93, 94, and 95 resins is Polyamide Resin No. 100. This is discussed in detail below.

Other valuable plasticizers include Abalyn (Hercules), the Aroclors (Monsanto), castor oil, most ester plasticizers including dibutyl phthalate and dibutyl sebacate, Dutrex 20 and 25 (Shell), and many of the Flexols (Carbon and Carbide).

Compatibility Relationships of Polyamide Resin No. 100 with Other Polyamide Resins

Polyamide Resin No. 100 plasticizes the nos. 90, 93, 94, and 95 resins and at the same time extends their sealing ranges and increases their versatility as heat seal adhesives. Interestingly enough, the addition of large quantities of the no. 100 resin can be made without decreasing, to the expected degree, the melting point of the higher melting resin. Conversely, the addition of small quantities of the higher melting resins to the no. 100 resin serves to raise the melting point of the latter appreciably. In the same vein, large quantities of the no. 94 resin [melting point = 105°C. (min.)] can be added to the higher melting no. 90 resin [melting point = 173° C. (min)] without appreciably lowering the melting

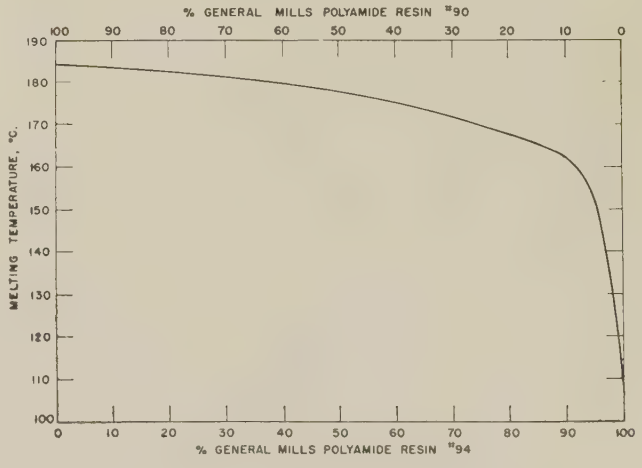


Fig. 4. Softening points of mixed Polyamide Resins Nos. 90 and 94

point of the latter resin. These relationships are demonstrated in Figs. 2, 3, and 4.

Compatibility Relationships of Polyamide Resin Suspensoids

Since polyamide resin suspensoids are cationic they are immediately compatible with virtually all cationically or nonionically stabilized emulsions, dispersions, and latices. As will be seen later they can also be made compatible with anionic latices.

The suspensoids are compatible with most of the commercial polyvinyl acetate emulsions. They serve to plasticize and to impart water resistance to the polyvinyl acetate films. Further, they reduce the marked tendency of the polyvinyl acetate to block. The polyvinyl acetate, on the other hand, improves the low temperature properties of the polyamide resin films, and broadens the temperature range over which heat seals will form.

Certain cationic latices such as Hycar OR-25 (Goodrich) and cationic Neoprene Latex (Du Pont) are compatible with the suspensoids, which plasticizes them and lowers their heat sealing range.

Compatibility of the suspensoids with widely used paper coatings such as clays, starches, and dextrines has already been discussed. If the dispersions are basic or anionic it is necessary to reverse the charge of the suspensoid in order to achieve compatibility. Charge reversal is accomplished by the rapid addition to the suspensoid of an excess of an anionic emulsifier such as Nacconol NRSF (National Aniline). Special Wheat Protein (General Mills, Inc.) serves as a stabilizer during this charge reversal process.

Once the charge of the suspensoid has been reversed it becomes compatible with a wide variety of anionically stabilized materials of interest to the paper converter

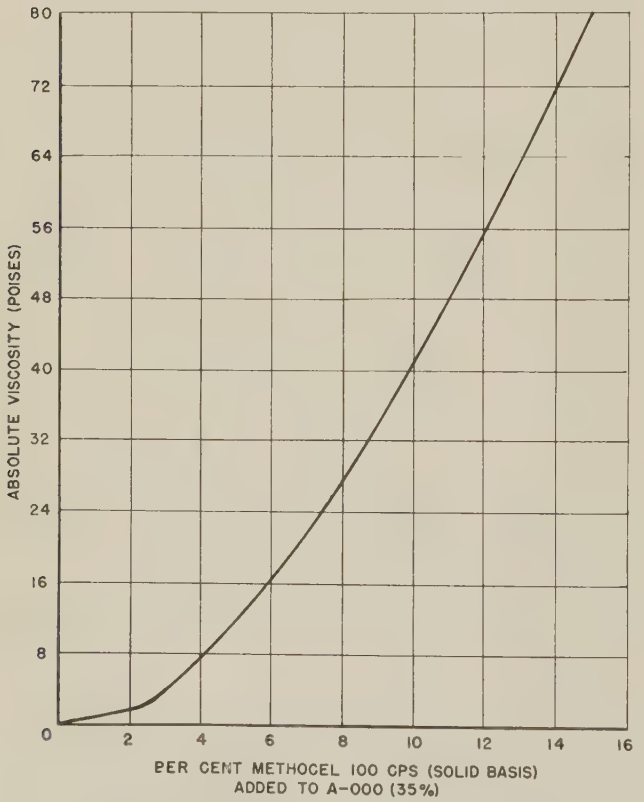


Fig. 5. Effect of Methocel 100 cp. on viscosity of Polyamide Resin Suspensoid A-000 (35% solids)

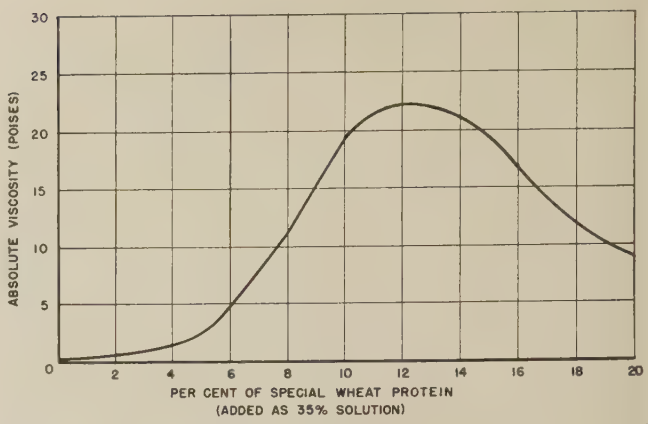


Fig. 6. Effect of special wheat protein on viscosity of Polyamide Resin Suspensoid A-000 (35% solids)

such as natural and synthetic latices (e.g., Dow Latex 512K); rosin emulsions such as Dresinol S-155 and Vinsol emulsion (Hercules); vinyl latices such as Geon latices (Goodrich), Vinylite W-125 (Bakelite), Saran latex (Dow); acrylic latices such as Polyco 422 and 423 (American Polymer Co.); and styrene latices such as Latex X-600 and 601-40 (Monsanto).

Casein solutions are compatible with the reversed charge suspensoid. On the other hand, Special Wheat Protein (General Mills, Inc.) serves to stabilize and increase the viscosity of the cationic suspensoid. Polyvinyl alcohol (Du Pont) and Methocel (Dow) are also good stabilizers and thickeners. Figures 5 and 6 show the effect of Special Wheat Protein and 100-cp. Methocel, respectively, on the viscosity of Polyamide Resin Suspensoid A-000, a composition based on the no. 95 resin. The Special Wheat Protein may be added as a 35 to 50% solution, whereas the Methocel is added as a 10% solution. Neither of these thickeners impairs the heat sealing properties of the suspensoid, and both contribute to the antiblocking properties.

Many resins and extenders which are not readily dispersed in water can be added to the suspensoids as solvent solutions. Rosin esters, Vistanex (Enjay), ethyl cellulose (Hercules or Dow), and many resins have been added as solutions in hydrocarbons or hydrocarbon-alcohol mixtures.

Solvents themselves are well tolerated by the suspensoids, and complete continuity is imparted to the films by butanol, either alone or with hydrocarbons and by methyl isobutylcarbinol, cyclohexanol, and butyl cellosolve. Many liquid plasticizers can be added directly with vigorous agitation.

COATING PROCEDURES

Laboratorywise, polyamide resin suspensoids and solutions may be coated successfully by the use of various coating bars such as Bird applicators and Mayer rods. Martinson and Mayer coaters and similar devices have also been used successfully.

For commercial hot melt coating, roll or transfer devices are widely used where the hot melt is applied to the web and then metered or smoothed. Cooling drums are used for rapid operation although the point must be made that the low operating temperature possible with polyamide resin and its quick setting properties make possible successful operation without cold rolls.

The rotogravure method, using recessed rolls to apply a predetermined amount of coating, can also be used with polyamide resin hot melts.

Reverse roll coating is probably the most popular procedure for coating webs with both the suspensoid and solvent solutions of the resin. A typical apparatus is well described in Fig. 19 of a TAPPI (*12*) monograph on coating machinery. Direct roll applicators with various doctoring devices have also been used. A device demonstrated in Fig. 6 of the above-mentioned monograph has been used successfully for polyamide resin solutions of high solids content and viscosity. Solutions here are best coated at temperatures in the region of 110°F. Some work has also been done with rotogravure printing of the solvent solutions. Air knife coating has been applied successfully to the resin suspensoids.

Both the resin solutions and suspensoids may be applied to surfaces by spraying.

The removal of water or solvent and the necessity for fusion of the films obtained from the suspensoid have already been discussed.

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Further Studies on the Alkaline Pulping of Scrub Oak

C. W. ROTHROCK, JR., and W. J. NOLAN

The effect of variations in per cent sulphidity of the cooking liquor and of additions of black liquor to the cooking liquor are shown. The range of 30 to 40% sulphidity is most suitable for producing an easily bleached pulp from scrub oak. Other necessary conditions are 16% total chemical and 35 grams per liter (expressed as Na_2O). Steam pressure should be 115 p.s.i.g., 1.0 hours to pressure and 1.5 to 2.0 hours at maximum pressure. Pulps require 7.0 to 8.0% available chlorine to produce a G.E. brightness of 75 in single stage. Additions of black liquor to the cooking liquor cause an immediate disappearance of almost half of titratable sulphide. The black liquor addition results in a milder cook with higher screenings than when no black liquor is used.

In a previous publication (*1*) the results of an investigation on the pulping of scrub oak (*Quercus laevis*) were presented. That work was done at a constant sulphidity of 25%. The pulping characteristics of the wood should be known for all possible conditions so that mill operation can be made as flexible as possible. It is for that reason that the present study was undertaken.

In this work the effect of changes in per cent sulphidity of the cooking liquor on the pulping characteristics of scrub oak was studied, and the corresponding changes in yield, screenings, bleach requirements, physical and chemical properties of the pulps were evaluated. In addition, the effect of black liquor additions to the cooking liquor before pulping, as normally practiced in mill operation, have been investigated.

It is to be noted that in the bleaching studies, the single stage method as described by TAPPI Standard T 219 m was used. It is planned to investigate multistage bleach methods and their effect on physical strength of the resultant pulps. This is to be reported at a later date.

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PROCEDURE AND PRESENTATION OF DATA

Pulping, washing, screening, bleaching, chemical and physical analysis, etc., were all carried out in the manner previously described (*1*). Two additional tests, not previously reported, were made on the pulps. These were determination of pentosan, carried out according to TAPPI Standard T 223 m, and alpha-cellulose by the TAPPI Standard T 429 m gravimetric method.

Duplicate pulping experiments were run preliminary to the sulphidity investigation, so that two complete cooking curves could be obtained under identical conditions for the purpose of determining accuracy of cooking data. Per cent of original wood removed, per cent screenings based on total yield, and liquor concentration were plotted against total cooking time. Smooth curves were drawn through the data points. Per cent wood removed was then read from the curve and compared to the actual data points. The data resulting from this duplication of cooks are presented in Table I. It should be noted that in all subsequent tables, the yield data listed are experimentally determined yield values rather than smoothed data read from the cooking curves.

The study of the effect of per cent sulphidity of the cooking liquor on the pulping reaction was carried out in the following order. First, per cent sulphidity was varied from 0.0 to 100% using other conditions found previously to be best (*1*). These conditions were 16% chemical, 35.0 grams per liter, 115 p.s.i.g., and 1.0 hours to maximum cooking pressure. Sulphidities studied under these conditions were 0.0, 15, 25, 30, 35,

40, 45, and 100%. Results of these cooks are presented in Table II as cook numbers 12-0-0 through 12-100-6.

Second, at the same 16% chemical and 115 p.s.i.g. steam pressure, concentrations of 20 and 50 grams per liter were used, each at 30, 35, and 40% sulphidity. These data are tabulated as cook numbers 18-30-0 through 18-40-6 and 6-30-0 through 6-40-6, respectively, in Table II.

Finally, per cent chemical was increased to 18% and sulphidities of 30, 35, and 40%, each at liquor concentrations of 20, 35, and 50 grams per liter, were used and the effects on the resultant pulps were determined. These data are tabulated in Table II as cook numbers 19 30-0 through 21-40-6. It is to be noted that in one case, the 20-25-0 through 20-40-6 series, about 25% sulphidity was used instead of the usual 30%. This was done to give a wider spread of sulphidity for comparison to the corresponding series of 16% chemical and 35 grams per liter.

A study of the effect of initial liquor concentration and per cent chemical on the rate of pulping was made using the data in Table II. The results of this study are tabulated in Table II-A. Each value listed for per cent yield and per cent screenings is the average of the 30, 35, and 40% sulphidity cooks for that specific cooking time. This procedure was considered justifiable as these sulphidities are in the range where the minimum effect of small changes in sulphidity is realized.

The above pulping experiments were carried on over an extended period. From time to time check cooks were run to determine the effect, if any, of chip age on the pulping characteristics of the wood. Chips had been dried immediately after chipping and were stored in an air-dry condition in well-ventilated bins. The results of these pulping experiments at chip ages of 0.0, 6, and 15 months are listed in Table III.

The sulphidity studies employed small size chips, produced in a brush hog, as material to be pulped. These chips consisted of the wood fraction passing through $^3/_4$-inch screen and retained on a $^1/_4$-inch screen. It seemed desirable to know whether these data would be applicable to mill size chips. For that reason a few oak logs were chipped at the Hudson Pulp and Paper Co., Palatka, Fla., and the commercial size chips were pulped and compared with the small chips cooked under identical conditions. These data are presented in Table IV.

Black liquor dilutions of the strong white liquor before pulping is common procedure in commercial pulping. Therefore, it is necessary that the effects of these additions be known for the pulping of the scrub oaks. A restricted study of the black liquor additions was made and the results are shown in Table V. Additions of 50 and 66.6% black liquor in the cooking liquor were made at diluted concentrations of approximately 35 grams per liter. The active alkali was 16% of the wood and cooking pressure was 115 p.s.i.g., 1.0 hour to maximum pressure. Calculated sulphidities were 25.0, 30.0, and 40.0%, based on the active alkali in the diluted cooking liquor.

Table V showed that when the diluted cooking liquor was titrated the per cent sulphidity determined by analysis did not correspond to the calculated value. Some further work was done on this discrepancy, wherein mixtures of white liquor and black liquor, both of whose analyses were known, were titrated at different time intervals after mixing. A comparison of calculated values and values determined by analysis after mixing is tabulated in Table VI.

Table I.[a] **Duplicate Cooking Data for Accuracy Checks**

Liquor concentration, g. Na_2O/l.	*Sulphidity, %*	*Total cooking time, hr.*	*Total yield, % of original wood*	*Total yield, read from cooking curves*	*Screenings, % of total yield*	*Bleach requirement, % available chlorine*
35.2	39.4	0	...	...	...	...
11.3	66.4	1.0	53.2	53.5	13.6	...
8.8	88.3	1.5	49.6	48.5	2.4	7.6
7.8	103.3	2.0	46.7	47.0	1.2	...
7.0	104.2	2.5	46.6	46.0	0.9	...
6.7	106.6	3.0	47.3	46.0	0.5	8.3
6.2	113.0	3.5	45.7	46.0	0.5	8.5
35.0	39.9	0	...	...	...	...
11.6	63.8	1.0	54.1	53.5	16.8	...
7.4	75.7	1.5	48.1	48.5	2.4	9.7
6.3	89.0	2.0	47.0	47.0	1.4	...
6.6	102.1	2.5	46.6	46.0	0.6	...
5.8	105.1	3.0	45.9	46.0	0.3	9.3
5.9	110.2	3.5	45.2	46.0	0.2	9.5

[a] 16% chemical (basis dry chips) used throughout. Maximum cooking pressure 115 p.s.i.g.; 1.0 hour to maximum pressure.

DISCUSSION

Accuracy of Data

Table I shows the cooking data for two sets of cooks carried out under identical cooking conditions. Yield and screenings data are reported to the nearest tenth of 1%. It can be seen by inspection that the 3-hour cook in the first set of cooks is not in line with the rest of the data. A smooth curve, drawn through the six data points, corrects for such errors. However, the nature of the complete procedure introduces small experimental errors which cannot be avoided. Moisture determination and weighing of chips, analysis and measurement of cooking liquor, moisture determination, and weighing of wet pulps (80% or more moisture content), etc., introduce small unavoidable errors. For that reason it is felt that yield values should not be relied on any closer than the nearest one half of 1%.

The tabulated values for per cent screenings at 1.0 and 1.5 hours' cooking time agree more closely than could be expected. Generally in such early stages of the pulping reaction the pulps contain large percentages of screenings. These screenings, by even the slightest mechanical handling, may be broken down partially into material which will pass through the flat screen. The amount of these screenings which convert into accepted fiber depend on the amount of handling and the cut of the screen plate. In this work a 10-cut screen plate was used. Mechanical handling was avoided by using lightly pressed pulp before screening so that adherence of fibers into balls would be avoided. This light pressing (about 80 to 82% moisture) avoided breaking up of screenings but made moisture determination for yield determination difficult. The moisture content listed above is slightly higher than the 75% used in previous work (*1*) as it was found that the lower moisture content occasionally caused balling of cooked fibers which were retained on the screen.

It seems, from the above considerations, that some standard method should be devised for determination of screenings in pulps. Probably some standard of disintegration before screening should be set up as moisture determination of very wet, raw pulps is difficult.

Effect of Sulphidity in Pulping at 16% Total Chemical and 35 Grams per Liter

The data in Table II cover the complete range of sulphidities studied. The results of only the 1.5 and 3.5-hour cooks are presented, as space requirements for presentation of all six cooks in each series would be prohibitive. All numerical values are observed rather than smoothed data.

The pulping experiments reported previously (*1*) showed that, at 25% sulphidity, 16% chemical and 35 grams per liter (both values expressed as Na_2O) were the optimum conditions for the cooking liquor when 115 p.s.i.g. steam pressure was used. Therefore the complete range of sulphidities was studied under these conditions. The sulphidities employed were 0.0, 15, 25, 30, 35, 40, 45, and 100% of total alkali. (It is noted that per cent sulphidity of white liquor is reported as per cent of total alkali. Per cent sulphidity of black liquor is expressed as per cent of active alkali.)

Yield of Pulp and Screenings. The data showed, from a yield standpoint, that the best range of sulphidities was between 25 and 45%. Yield at 0.0% sulphidity and 1.0 hours' cooking was 59.5%. Yield decreased with sulphidity to 52.5 to 53.5% for 1.0-hour cooks at sulphidities of 25 to 40%. The corresponding yield value for 45% sulphidity was 56.0%, while at 100% sulphidity yield was 60.5.

Per cent screenings for these same pulps were as follows: at 0.0% sulphidity—89%; 15% sulphidity—50%; 25% sulphidity—39%; 30% sulphidity—19%; 35% sulphidity—20%; 40% sulphidity—17%; 45% sulphidity 17%; 100% sulphidity—89%.

At the end of 1.5 hours' cooking time Table II shows that the highest screened yields and lowest screenings came at 30 to 45% sulphidity. As the cooking time increased further, yield and screening values tended to become constant, independent of per cent sulphidity except, of course, for the 100% sulphidity cooks. From the standpoint of yield and screenings alone, therefore, this series of cooks indicated that 30 to 45% sulphidity was the optimum range.

Space limitations prohibit the submission of a graphical presentation of any of the cooking data in this paper. However, curves of wood removed, per cent screenings, and chemical concentration of the cooking liquor, when plotted against cooking time, follow the same general form as in Fig. 1 of the first publication in this series (*1*). Superimposing the few data points given in Table II on that figure will give one a fair idea of the general trends throughout the cook.

Chemical Analysis of Pulps. Consideration of the effect of sulphidity on chemical analysis of the 3.5-hour cooks leads to the following facts. At 25% sulphidity alpha-cellulose was 81.0%; at 30% sulphidity, alpha was 79.8%; at 35% sulphidity, 84.2%; at 40% sulphidity, 87.2%; at 45% sulphidity, 82.1%. Pentosan content of pulps produced in the range of 25 to 45% sulphidity was quite uniform varying between 19.0 and 20.0%. Lignin content decreased from 2.2% at 25% sulphidity to 1.7 and 1.5% at 35 and 40% sulphidity, respectively. At 45% sulphidity lignin did not increase but remained at 1.4%. At 100% sulphidity lignin was 6.2%. It can be seen that 35 to 40% sulphidity produced the best pulps from consideration of chemical analysis. However, the superiority of these two concentrations in the over-all range 25 to 45% sulphidity is not very great.

Bleachability of Pulps. Bleach requirements in the series of cooks employing 16% chemical on a basis of wood showed a peculiar trend. As sulphidity increased from 0.0 to 25% bleach requirements decreased. From 30% sulphidity up through 45% bleach requirements increased in spite of the fact that lignin continued to decrease to as low as 1.4%. At 25% sulphidity lignin content was 2.3%, and bleach requirement was 7.0%. At 40% sulphidity lignin content was only 1.5% but bleach requirement was 8.5. At 45% sulphidity lignin was 1.4, and bleach requirement was 9.4. This trend indicates that the higher sulphidities may form some compound with lignin which is difficult to oxidize. It will be interesting to determine whether this trend in bleachability has a harmful effect on strength characteristics of bleached pulps.

Permanganate numbers were determined for most pulps whose bleach requirements had been evaluated but no correlation could be found between permanganate number of bleach requirement. This lack of correlation is not entirely due to the fact that sulphidity of white liquor varied from one set of cooks to another. In several sets of cooks, three bleachabilities and corresponding permanganate numbers were determined but no trend toward a straight line relationship could be found.

Physical Strength of Pulps. The physical strength properties of pulps produced at 16% chemical, 35 grams per liter concentration, and varying sulphidity are shown in Table II, along with strength values for other concentrations and 18% total chemical values. These values are for a Canadian Standard freeness of 500. The values shown are not indicative of the true potential strength values of any of the pulps. In all cases maximum tear occurred in the neighborhood of 300 freeness while burst, breaking length, and fold values increased continuously with beating time.

An average of the strength values for all conditions listed in Table II was made in order to show the difficulties involved in an evaluation of one pulp against another. These averages cover 16 and 18% chemical, 20, 35, and 50 grams per liter, and 25 through 45% sulphidity. The maximum tear value realized was 90, with an average of the maxima for all cooks of 83.8. Greatest deviation from this average was +7.0 and −14.0%. In breaking length the maximum value, at about 100 freeness, was 10,100 meters with an average for the maxima of all cooks at 9500. Greatest deviation was +6.3 and −7.4%. In burst factor the maximum at about 100 freeness was 64 with an average for all the maxima of 58.2. Deviation from the maxima was +10.3 and −12.0%. In double folds, the most erratic of all physical strength factors, the maximum value realized was 1070 with an average for the maxima of all cooks of 724. Here the deviation from the average was +47.7 and −28.0%. All numerical values of physical strength are expressed in units called for by TAPPI Standard T 220 m.

It can be seen that, with the small differences in all strength values except fold, evaluation of the relative merits of the pulps on a strength basis was difficult. The method used consisted of comparison of the plotted strength data of each pulp with all the others by superimposing one chart on another and examining the pair

under a strong light. Relative differences in the four strength curves determined which was the better. By elimination, the pulps were listed in the order of their relative strengths. Two observers used this method of visual judgment and comparison of results found the two listings to agree very closely.

It was found, in the series of pulps produced with 16% total chemical and 35-gram per liter concentration, that the strongest pulps were produced at sulphidities between 30 and 40%. In this range, no significant differences in the four strength characteristics of the three pulps could be detected. Pulps cooked 1.5 hours were definitely stronger than those pulped 3.5 hours. This higher strength in the earlier cooks was found to be true of the entire series. Sulphidities of 25 and 45% were inferior in strength to the 30 through 40% sulphidities. At 1.5 hours' cooking time the 25 and 45% sulphidity cooks were equal in strength but at 3.5 hours' cooking time the 45% sulphidity retained more of the pulp's strength than 25%. This point seems reasonable as the higher sulphidity should tend to retard degradation of the fibers.

Pulps produced with 15% sulphidity were much lower in strength than those produced with more sulphide. The cooks using no sulphide were much weaker than when only 15% sulphidity was used. The cooks employing 100% sulphidity were so raw that sheets could not be made.

Therefore, for highest strength pulps, sulphidity should be between 30 and 40% when 16% total alkali and 35 grams per liter are employed. Cooks of less than 3.5 hours' duration should be employed if highest strength is to be realized.

Effect of Sulphidity on Pulping at 16% Total Chemical, 20 and 50 Grams per Liter

The data discussed above indicated that the best all-round sulphidity should occur between values of 30 and 40%. For that reason, the effects of varying the liquor concentration of active alkali between 20 and 50 grams per liter in this range of sulphidity were investigated. The data for this phase of the work are presented in Table II, listed as cook nos. 18-30-0 to 18-40-6 and 6-30-0 to 6-40-6 for 20 and 50 grams per liter, respectively. The shortest cooking time used in pulp analysis of the 20-gram per liter cooks was 2.0 hours as the 1.5-hour cooks were too raw containing 5.9 to 10.6% screenings. The 50-gram per liter cooks, however, utilized the 1.5 hours' cooking time as the screenings here were lower, 2.8 to 4.0% of the total yield. This variation of cooking time need not interfere with direct comparison of pulps as the additional half hour of cooking time for the low concentration produces practically the same degree of pulping as the shorter cooks at higher concentrations, as is borne out by a comparison of yield and screenings data.

The effect of sulphidity changes in this series of pulps should be minor since per cent sulphidities were in the optimum range and neither a large insufficiency nor excess of sulphide was studied. The main variations in pulps should be due to initial concentration variations rather than variations in sulphidity. This was found to be true.

Yield of Pulp and Screenings. There is a slight indication from the yield and screenings data that the 40% sulphidity does not pulp quite as drastically as the 30 and 35% values. However the trend is not sufficient to be of real significance.

Table II-A is a compilation of all yield and screenings data as affected by changes in concentration. Since only slight effects were found from sulphidity changes in the range of 30 to 40%, yield and screenings data are the average for three cooks in this sulphidity range. The effect of variations of initial concentration is very marked and in the direction to be expected. In the first hour of pulping higher concentration produces lower yields with correspondingly lower screenings. At 1.5 hours this effect diminishes but is still apparent. At 3.5 hours all concentrations produce pulps of practically the same yield and screenings. For rapid pulping 35 to 50 grams per liter should be used. Since there is no difference in the later cooks, the higher concentrations, from a yield standpoint, should still be used as small volumes of black liquor will be involved.

Chemical Analysis of Pulps. Lignin determinations on the 20-gram per liter cooks were the only chemical analyses made on this series of pulps. Changes of sulphidity in the range studied appear to have no appreciable effect on lignin content, within the limits of the method of lignin analysis. At 2.0 hours' cooking time the average lignin content of the three pulps cooked at 30, 35, and 40% sulphidities was 2.4% with a deviation of +0.3 to −0.4%. At 3.5 hours the average lignin content was 1.6% with deviations of +0.4 and −0.5%. Concentrations of 20 grams per liter required $^1/_2$ hour longer in the early stages of pulping to attain the same lignin removal from screened pulp as 35 grams per liter. At 3.5 hours concentration seemed to have no effect.

Bleachability of Pulps. Concentration in this narrow range of sulphidities has more effect than sulphidity. As concentration of active alkali increases, bleach requirements of the pulps decrease somewhat. On an average, over the range of sulphidities studied, 2.0 to 2.5% difference in bleach was required in the concentration range of 20 to 50 grams per liter. It appears that 35 or 50 grams per liter should be used for best bleach characteristics.

Physical Strength of Pulps. The physical strength values of these pulps, cooked with sulphidities of 30, 35, and 40% and liquor concentrations of 20 and 50 grams per liter, all at a total chemical of 16%, are listed in Table II. These strengths, as mentioned earlier, are taken at a Canadian Standard freeness of 500 and are not representative of the true potential strength characteristics of the pulps. Cross checking of the strength values of the various pulps produced the following observations.

At 20-gram per liter concentration the strength development of pulps cooked at the three sulphidities was almost identical. It was impossible, by visual comparison of one set of curves to another to say that any one pulp was better in strength than another. Even the freeness development curves coincided. There was no distinguishable difference in strengths of pulps cooked at 2.0 hours from those produced at 3.5 hours.

At 50 grams per liter no effect of sulphidity could be noted. Differences in strength between pulps cooked at 1.5 hours and those cooked at 3.5 hours were very marked, The increased cooking time caused a loss in development of all strength characteristics, much more

Table II. Effect of Sulphidity on Pulping of Scrub Oak

(Max. cooking pressure 115 p.s.i.g., 1.0 hr. to max. pressure throughout)

Cook no.	Active alkali, % Na_2O to wood	Liquor concentration, g. Na_2O/l.	Sulphidity, %	Total cooking time, hr.	Total yield, % of original wood	Screenings, % of total yield	Lignin in pulp, %	Permanganate no.	Bleach requirement, % available chlorine	Beating time to 500 C.S.F., min.	Strength data at 500 Canadian Std. freeness: Breaking length	Tear factor	Burst factor	Double folds
12- 0-0	16	35.8	0	0	...	...	27.0	...	...	...	...	...	...	...
12- 0-2	16	10.0	0	1.5	51.0	13.4	7.6	23.7	26.0	22	4230	64.4	15.8	12
12- 0-6	16	5.3	0	3.5	45.2	0.1	2.4	15.7	11.3	10	4620	64.0	19.5	16
12- 15-0	16	35.0	14.2	0	...	...	27.0	...	...	...	...	...	...	...
12- 15-2	16	8.0	7.5	1.5	48.9	2.4	2.2	16.6	11.3	13	6850	71.7	23.2	10
12- 15-6	16	3.4	16.3	3.5	45.2	0.0	1.3	11.7	8.7	12	5050	61.2	22.5	20
12- 25-0	16	35.1	24.8	0	...	...	27.0	...	...	...	...	...	...	...
12- 25-2	16	8.4	44.0	1.5	48.3	6.0	...	...	8.0	14	5980	79.0	24.6	42
12- 25-6	16	5.5	79.2	3.5	45.1	0.8	2.2	...	7.0	14	6000	74.3	26.0	40
12- 30-0	16	35.5	30.4	0	...	...	27.0	...	...	...	...	...	...	...
12- 30-2	16	7.7	53.3	1.5	47.3	2.1	...	14.4	8.4	9	5680	77.0	30.0	45
12- 30-6	16	4.8	90.5	3.5	44.2	0.4	2.2	12.4	7.9	5	5400	72.5	24.5	30
12- 35-0	16	35.1	34.3	0	...	...	27.0	...	...	...	...	...	...	...
12- 35-2	16	6.9	51.9	1.5	47.5	1.9	2.6	14.6	8.4	7	5750	81.6	28.5	57
12- 35-6	16	5.4	104.2	3.5	45.3	0.2	1.7	12.6	8.9	7	5680	79.8	30.1	52
12- 40-0	16	35.2	39.4	0	...	...	27.0	...	...	...	...	...	...	...
12- 40-2	16	8.8	88.3	1.5	49.6	2.4	...	15.0	7.6	6	5470	80.4	28.7	50
12- 40-6	16	6.2	113.0	3.5	45.7	0.5	1.5	13.0	8.5	11	6120	76.8	25.2	62
12- 45-0	16	34.9	45.3	0	...	...	27.0	...	...	...	...	...	...	...
12- 45-2	16	6.4	89.1	1.5	47.8	1.3	2.2	14.8	9.3	6	5400	80.0	23.7	22
12- 45-6	16	5.1	111.6	3.5	45.7	0.1	1.4	12.6	9.4	13	5600	80.9	27.7	35
12-100-0	16	35.3	98.8	0	...	...	27.0	...	...	...	...	...	...	...
12-100-2	16	9.1	176.5	2.0	56.7	68.9	7.7	...	...	...	...	...	...	...
12-100-6	16	9.8	172.5	3.5	54.6	18.8	6.2	...	...	...	...	...	...	...
18- 30-0	16	19.8	30.3	0	...	...	27.0	...	...	...	...	...	...	...
18- 30-3	16	4.4	60.9	2.0	47.2	1.6	2.7	16.0	10.0	8	6020	76.2	25.6	54
18- 30-6	16	3.4	70.4	3.5	44.6	0.5	2.0	13.5	8.4	3	5430	69.4	25.8	30
18- 35-0	16	19.8	34.9	0	...	...	27.0	...	...	...	...	...	...	...
18- 35-3	16	3.7	19.2	2.0	47.7	1.5	2.0	16.9	11.3	5	5630	76.2	25.3	32
18- 35-6	16	3.3	106.8	3.5	44.9	0.4	1.1	13.9	10.1	3	5200	68.0	24.5	22
18- 40-0	16	19.9	40.4	0	...	...	27.0	...	...	...	...	...	...	...
18- 40-3	16	4.4	90.3	2.0	46.3	3.5	2.6	15.6	10.3	7	5320	74.7	27.9	33
18- 40-6	16	3.7	102.5	3.5	45.5	1.1	1.6	13.8	9.0	8	6000	75.2	29.0	48
6- 30-0	16	50.2	29.7	0	...	...	27.0	...	...	...	...	...	...	...
6- 30-2	16	10.8	77.7	1.5	46.3	2.8	...	15.2	7.3	11	5730	67.2	29.4	41
6- 30-6	16	7.4	99.0	3.5	45.1	0.4	...	13.2	6.9	15	5540	65.8	27.0	52
6- 35-0	16	49.9	35.1	0	...	...	27.0	...	...	...	...	...	...	...
6- 35-2	16	11.4	83.6	1.5	46.5	3.7	...	15.6	7.1	13	6320	82.8	30.7	80
6- 35-6	16	9.0	112.6	3.5	44.4	0.3	...	13.3	7.1	15	5520	70.5	26.0	40
6- 40-0	16	49.7	40.2	0	...	...	27.0	...	...	...	...	...	...	...
6- 40-2	16	10.8	96.0	1.5	47.3	4.0	...	15.2	7.8	16	6400	80.8	33.2	80
6- 40-6	16	9.1	103.1	3.5	45.2	0.5	...	12.9	7.8	16	6820	75.0	30.0	50
19- 30-0	18	20.0	30.5	0	...	...	27.0	...	...	...	...	...	...	...
19- 30-3	18	5.7	51.9	2.0	47.2	3.1	...	16.6	8.4	11	5940	78.7	24.8	52
19- 30-6	18	4.2	...	3.5	44.9	0.4	...	13.6	6.9	10	6130	80.4	27.2	52
19- 35-0	18	19.8	34.9	0	...	...	27.0	...	...	...	...	...	...	...
19- 35-2	18	8.3	30.1	1.5	48.0	5.9	...	18.2	10.5	15	6130	75.2	26.1	44
19- 35-6	18	...	...	3.5	44.1	0.3	...	13.4	7.3	12	6350	74.8	27.7	70
19- 40-0	18	19.8	39.9	0	...	...	27.0	...	...	...	...	...	...	...
19- 40-3	18	...	...	1.5	48.8	5.9	...	15.7	10.4	...	...	...	...	...
19- 40-6	18	...	...	3.5	45.5	0.2	...	13.0	6.6	9	6000	77.0	28.1	62
20- 25-0	18	34.6	23.6	0	...	...	27.0	...	...	...	...	...	...	...
20- 25-2	18	10.0	37.8	1.5	48.1	5.2	...	...	7.3	14	5780	80.1	27.5	62
20- 25-6	18	6.5	30.9	3.5	45.1	0.3	...	...	6.1	12	6030	73.5	27.7	48
20- 35-0	18	35.2	35.0	0	...	...	27.0	...	...	...	...	...	...	...
20- 35-2	18	8.5	46.4	1.5	48.1	3.8	1.0	15.6	8.1	11	6220	78.0	27.7	48
20- 35-6	18	6.0	85.9	3.5	44.6	0.4	0.5	12.7	7.6	11	6020	71.4	27.5	46
20- 40-0	18	35.3	39.8	0	...	...	27.0	...	...	...	...	...	...	...
20- 40-2	18	9.9	76.7	1.5	47.2	3.1	...	16.1	7.0	...	...	...	...	...
20- 40-6	18	7.3	104.4	3.5	44.2	0.3	...	12.7	6.8	14	6400	69.3	29.2	76
21- 35-0	18	50.2	34.5	0	...	...	27.0	...	...	...	...	...	...	...
21- 35-2	18	...	...	1.5	47.5	4.2	...	15.2	7.5	12	6000	72.3	26.3	63
21- 35-6	18	...	...	3.5	44.6	0.5	...	13.2	7.6	17	6030	67.3	25.9	52
21- 40-0	18	50.5	40.4	0	...	...	27.0	...	...	...	...	...	...	...
21- 40-2	18	13.1	88.4	1.5	46.9	2.5	...	15.0	7.4	12	6270	77.2	27.7	107
21- 40-6	18	9.3	111.5	3.5	44.8	0.3	...	12.9	7.5	17	6210	71.7	28.3	66

marked than in the 35-gram per liter series, reported above.

A comparison of the pulps cooked in this sulphidity range shows that sulphidity has little or no effect on strength development. Expansion of the sulphidity range in the 20 and 50-gram per liter range would undoubtedly result in the same conclusions as those discussed under the 35-gram per liter cooks.

The effect of concentration in this narrow range of sulphidities is as follows. In early cooks, 20-gram per liter liquors produce pulps of the same over-all strength as liquors of 35 grams per liter. Liquors of 50-gram per liter produce pulps with somewhat inferior strength characteristics.

Pulps produced from liquors containing 20 grams per liter and a cooking time of 3.5 hours are slightly superior to those cooked with 35-gram per liter liquor. This is due to the fact that cooking time has very little effect

Table II-A. Effect of Initial Concentration and Per Cent Chemical on Rate of Pulping

(Data are average of the 30, 35, and 40% sulphidity cooks)

Total cooking time, hr.	*Active alkali, % Na_2O to wood*	*Liquor concentration, g. Na_2O/l.*	*Total yield, % of original wood*	*Screenings, % of total yield*
1.0	16.0	20.0	59.2	64.8
1.0	16.0	35.0	53.4	18.7
1.0	16.0	50.0	49.2	12.0
1.0	18.0	20.0	57.3	60.7
1.0	18.0	35.0	54.7	27.3
1.0	18.0	50.0	51.5	12.2
1.5	16.0	20.0	50.0	7.2
1.5	16.0	35.0	47.4	2.6
1.5	16.0	50.0	46.6	3.3
1.5	18.0	20.0	49.0	7.3
1.5	18.0	35.0	47.9	4.2
1.5	18.0	50.0	47.2	3.5
3.5	16.0	20.0	44.8	0.5
3.5	16.0	35.0	45.7	0.2
3.5	16.0	50.0	45.0	0.5
3.5	18.0	20.0	45.2	0.5
3.5	18.0	35.0	44.8	0.5
3.5	18.0	50.0	44.9	0.5

on the strength of the very mild cooks while 35-gram per liter liquor causes the strength of pulps to decrease slightly. Liquors of 50 grams per liter cause all strength values to deteriorate sharply with time, resulting in definitely inferior pulps at 3.5 hours' total cooking time.

From consideration of pulp strength above it may be said that cooking liquors of 20 grams per liter are slightly superior to 35 grams per liter while 50 grams per liter are definitely inferior. Liquors of 35 grams per liter appear to be the best choice over-all when 16% chemical is used.

Effect of Sulphidity on Pulping at 18% Chemical at 20, 35, and 50 Grams per Liter. The cooking, bleaching, and physical strength values of this series of cooks are reported in Table II under cook nos. 19-30-0 through 21-40-6. It had been reported in the earlier publications (*1*) that 18% chemical was in excess and would tend to overcook. Pulps reported here were not appreciably overcooked. An inspection of the chemical concentration data for the cooks listed in Table II shows that the liquors using 18% chemical maintained a somewhat higher concentration throughout the cook than those using 16% chemical. However, these differences in concentration occurred at very low values (about 10 grams per liter or less) where reaction velocity is low. If cooking pressure were increased, stimulating reaction velocity and consumption of chemical, 18% chemical would definitely overcook the pulps. Subsequent discussion will point out that more than 16% is in excess.

It should be explained that in the series of cooks using 18% chemical and 35 grams per liter liquor concentration, a slightly wider spread of sulphidities, 25, 35, and 40%, were used. This was done to produce a somewhat closer comparison with the original wide range of sulphidities at 16 per cent chemical and 35 grams per liter without resorting to a large number of additional cooks. At this stage of the investigations over 150 individual cooks had been made and this decision on change in sulphidity can be understood.

Yield of Pulp and Screenings. No significant changes in either yield of pulp or screenings could be detected in this series of cooks from the corresponding cooks made at 16% chemical. As pointed out above, concentration differences in the two series were too slight to create appreciable differences in reaction rate. Sulphidity changes in this range had no tangible effect.

Chemical Analysis of Pulps. No chemical analysis data were determined on the pulps other than lignin in the pulps produced at 35 grams per liter. Per cent lignin in these pulps was almost identical with those resulting from 16% chemical.

Bleachability of Pulps. There is some indication from a comparison of the bleachability of the pulps produced at 16 and 18% chemical that the use of more chemical might be expected to slightly reduce bleach requirements. Increased concentration of cooking liquor appears to tend toward producing an easier bleaching pulp. Change in sulphidity in the range studied has negligible effect.

All pulps in this range of sulphidity and concentrations, considering that bleachabilities were determined in a single-stage process, were so low in bleach requirements that changes in pulping variables could not be expected to cause large changes in bleachability. If bleached in a multistage process, requirements in the whole range would probably vary between 4.4 and 7.1%.

Physical Strength of Pulps. The physical strengths at 500 freeness listed in Table II for this series are, as mentioned earlier, not representative of the potential strength of the pulps. Maximum strengths in the series developed at the same beating times as pulps previously discussed.

The strength characteristics of the pulps in this series showed the same relationship to the cooking variables as the pulps where 16% chemical was used. At 20-gram per liter concentration, physical strength was practically independent of per cent sulphidity and cooking time. These pulps were the strongest in this series and slightly superior to the best pulps at 16 grams per liter.

Pulps produced from 35 and 50-gram per liter concentration liquor were not appreciably affected by changes in the narrow range of sulphidity studied but strength values decreased with cooking time. Liquors of 35 grams per liter produced pulps stronger than 50 grams per liter but weaker than 20 grams per liter.

Following is a list, in order of physical strength, of all the pulps studied:

1. 18% total alkali, 20 g. per l., 30–40% sulphidity.
2. 16% total alkali, 20 g. per l., 30–40 %sulphidity.
3. 16% total alkali, 35 g. per l., 30–40% sulphidity.
4. 18% total alkali, 35 g. per l., 25–30% sulphidity.
5. 16% total alkali, 50 g. per l., 30–40% sulphidity.
5. 18% total alkali, 50 g. per l., 30–40% sulphidity.

The greatest magnitude of change came between numbers 3 and 4 in the listing. From 1 through 3 the pulps were very similar in numerical strength values. The last two pulps listed are both designated as number 5 as their strength values were very similar and definitely the poorest of all pulps from a strength standpoint.

Effect of Chip Storage Time on Pulping

A large supply of scrub oak logs, sufficient for the complete series of experiments, was chipped by the no. 0 Mitts and Merrill hog. Chips were air dried and stored in well-ventilated bins. The hog produced small-sized chips, the fraction passing $^3/_4$-inch screen opening and retained on $^1/_4$-inch screen being considered acceptable.

Table III.[a] Effect of Chip Storage Time on Pulping

Chip age, mo.	Liquor concentration, g. Na_2O/l.	Sulphidity, %	Total cooking time, hr.	Total yield, % of original wood	Screenings, % of total yield	Bleach requirement, % available chlorine
0	35.3	24.2	0	...	...	...
0	11.8	22.9	1.0	55.1	34.4	...
0	8.2	35.8	1.5	47.1	1.6	7.5
0	6.9	45.0	2.0	46.6	0.4	...
0	6.0	54.3	2.5	46.3	0.3	...
0	5.1	38.4	3.0	45.3	0.1	6.1
0	4.0	23.0	3.5	45.3	0.0	5.9
6	35.0	25.8	0	...	...	...
6	9.4	39.2	1.0	52.9	16.0	...
6	6.9	39.7	1.5	47.5	2.0	...
6	5.4	47.4	2.0	46.6	1.3	...
6	5.4	61.0	2.5	46.9	0.2	...
6	4.2	...	3.0	45.7	0.4	...
6	4.7	67.7	3.5	45.8	0.3	...
15	35.1	24.8	0	...	...	...
15	11.9	...	1.0	54.8	39.2	...
15	8.4	44.0	1.5	48.3	6.0	8.0
15	7.0	55.7	2.0	46.6	3.4	...
15	6.2	56.4	2.5	45.5	2.0	...
15	6.1	68.9	3.0	45.7	0.9	...
15	5.5	79.2	3.5	45.1	0.8	7.0

[a] 16% chemical (basis dry chips) used throughout. Maximum cooking pressure 115 p.s.i.g., 1.0 hour to maximum pressure.

The pulping program was known to be long range requiring periodic checks on the pulping characteristics of the wood as a function of age. It was not expected that age of chips would create another cooking variable since chips were dried before storage. Results of these check cooks are shown in Table III. The yield and bleaching data do not show any appreciable effect due to age of chips. There is a slight indication that the older chips may produce somewhat higher screenings than new chips but the data show that direct comparison of cooks throughout the duration of the experiments may be made.

Effect of Chip Size on Pulping

Some of the 15-month-old chips were rescreened and the fractions between $^1/_2$ and $^1/_4$-inch screen opening were saved. Additional new logs were debarked and chipped in a standard Carthage chipper at the Hudson Pulp and Paper Co., Palatka, Fla. These chips were screened, and the fraction retained between 1 and $^1/_2$-inch screen opening was saved for comparison with small-sized chips as a source of pulp. Cooking data for these comparison cooks are shown in Table IV.

Total yield and screenings of the mill-size chips were higher than those for small-sized chips but bleach requirements of the screened pulp were lower. These facts may be explained by converting total yield to screened yield. Screened yield from the large chips at 1 hour cooking time was 33.4%, reached a maximum of 46.7% at 2.5 hours, then decreased gradually to 45.3 at 3.5 hours' cooking time. The small chips at 1 hour total time had a screened yield of 45.0, reached a maximum of 46.9 at 1.5 hours, then decreased steadily to 45.1% at 3.5 hours.

Table IV.[a] Effect of Chip Size on Pulping

Chip size, screen opening, in.	Liquor concentration, g. Na_2O/l.	Sulphidity, %	Total cooking time, hr.	Total yield, % of original wood	Screenings, % of total yield	Bleach requirement, % available chlorine
$-1 + ^1/_2$	34.8	38.7	0	...	...	...
$-1 + ^1/_2$	11.6	40.2	1.0	57.6	42.1	...
$-1 + ^1/_2$	8.7	61.4	1.5	48.9	11.0	7.6
$-1 + ^1/_2$	8.0	96.7	2.0	48.5	6.7	...
$-1 + ^1/_2$	7.2	103.7	2.5	48.2	3.2	...
$-1 + ^1/_2$	6.8	111.2	3.0	46.8	2.5	6.7
$-1 + ^1/_2$	6.6	113.2	3.5	45.8	1.1	6.9
$-^1/_2 + ^1/_4$	35.0	39.9	0	...	...	...
$-^1/_2 + ^1/_4$	11.6	63.8	1.0	54.1	16.8	...
$-^1/_2 + ^1/_4$	7.4	75.7	1.5	48.1	2.4	9.7
$-^1/_2 + ^1/_4$	6.3	89.0	2.0	47.0	1.4	...
$-^1/_2 + ^1/_4$	6.6	102.1	2.5	46.6	0.6	...
$-^1/_2 + ^1/_4$	5.8	105.1	3.0	45.9	0.3	9.3
$-^1/_2 + ^1/_4$	5.9	110.2	3.5	45.2	0.2	9.5

[a] 16% chemical (basis dry chips) used throughout. Maximum cooking pressure 115 p.s.i.g., 1.0 hour to maximum pressure.

From screened yield data it is apparent that the smaller chips presented much more surface area for reaction with the chemical during the first 2.0 to 2.5 hours of cooking time. This surface area was made up of the surface of the uncooked chip plus the surface of all the loose fibers or fiber bundles already freed from the chip. Since the small chips presented more surface for reaction, the over-all delignification of the screened pulp at the same chemical consumption would be less for the small chips. In other words, the large chips are employing fewer fibers to produce the same drop in concentration caused by a relatively greater weight of fibers from the small chips during the first half of the cook. This would account for the higher bleach requirements of the pulps made from small chips.

It should be pointed out that, if short-time pulping is contemplated, mill-size chips should be cooked 0.5 to 1.0 hours longer than small chips. For example, a 1.5-hour cook on small chips would require between 2.0 and 2.5 hours for mill chips. A 2.0-hour cook on small chips would correspond to a cook of about 2.75 hours on large chips. At 3.5-hours both cooks should yield about the same type pulp.

Physical strength data were determined for pulps produced from both mill-size and small chips at 3.5 hours' cooking time. Breaking length and fold, plotted as a function of beating time, were almost identical. Pulp from mill-size chips developed a higher maximum tear, 93, about 10% higher than pulp from small size chips, but required 55 minutes' beating time to attain this value as compared to 30 minutes for the smaller chips. A bursting strength of 20 on the unbeaten pulp from mill chips was realized corresponding to 15 for the unbeaten small chip pulp. At 45 minutes the two curves for burst against beating time had joined at a value of 51. Freeness developed more slowly on the large size, requiring 30 minutes to reach 400 Canadian Standard freeness, compared to 21 minutes for the other pulp.

Effect of Black Liquor Additions to Cooking Liquors

Several experimental cooks were made in which black liquor was added to the white liquor before pulping. In some cases 50.0% of the total cooking liquor was black liquor, in others 66.6% of the total liquor was black liquor. In all cases per cent sulphidity and concentration were calculated on a basis of the mixed liquor. Black liquor was obtained from the Hudson Pulp and Paper Co., recovered from the kraft pulping of slash pine and containing approximately 18% solids.

Calculated sulphidities of 25, 30, and 40% sulphidities were used, with a chemical concentration of 35 grams per liter and a total alkali of 16%, based on dry wood. Yield, bleach, and chemical analysis data are shown in Table V.

Yield of Pulp and Screenings. The yield and screenings data show that neither variations in sulphidity nor per cent black liquor had any appreciable effect on rate of pulping. Black liquor additions apparently reduce the activity of the cooking liquor as yield and screenings values were higher than cooks where no black liquor had been added.

The discrepancies between calculated and actual values of sulphidity listed in the table probably account for the high screenings in the pulps. Actual values are those determined by titration, using TAPPI Standard T 625 m, Na_2S being found through the generation of H_2S. There appears to be a very considerable loss of sulphide as soon as the white and black liquors are mixed.

Chemical Analysis of Pulps. Lignin content of pulps was somewhat higher, 2.1 to 2.7% at 3.5 hours' cooking time, than corresponding values for cooks without black liquor, 1.5 to 2.2%. Alpha-cellulose values were quite similar to cooks in which no black liquor was used ranging between 78.3 and 85.8%. Pentosans, varying between 20.6 and 23.0%, showed no correlation with per cent black liquor used. They were higher than for pulps in which white liquor alone was used where pentosan content varied between 17.5 and 20.0%.

Bleachability of Pulps. Bleach requirements, with one exception, were quite similar to the values required by pulps cooked with white liquor. In one case, in which 25% sulphidity and 66.6% black liquor were used, over 11% bleach was required. Nine per cent bleach brought the pulp to a G.E. brightness of 73. Beyond this point increases in bleach did not raise the brightness even though 11.0% total bleach was used. The peculiar behavior of this one pulp cannot be explained.

Disappearance of Titratable Sulphide. The actual sulphidities of the cooking liquors listed in Table V showed definite disappearance of titratable sulphide between the time white and black liquors were mixed and the time the cooks were started. In all cases liquors were mixed the afternoon preceding the day the liquors were titrated and used. For that reason additional tests were made in which the liquors were titrated at definite time intervals after mixing. These data are presented in Table VI.

The sulphide content of the liquors dropped, within 1 hour after mixing from the calculated value to 17.3 to 22.0%. Loss in sulphide increased with amount of black liquor added. Elapsed time after mixing decreased the titratable sulphide especially at the higher percentages of black liquor. Active alkali is not appreciably decreased by the drop in sulphidity.

A further check on this loss of sulphide was made, eliminating as far as possible the time interval between mixing and analysis. White and black liquor were mixed and analyzed immediately. At the same time, the mixture was introduced into a digester charged with chips and the cooking cycle was begun. Liquor samples were taken every 10 minutes for the 60 minutes required to bring the digester to the full cooking pressure of 115 p.s.i.g. Six cooks were made, only one liquor sample being taken from each cook to avoid change in per cent chemical due to removal of liquor samples. Conditions of pulping were 50% black liquor, 35% calculated sulphidity, 16% total chemical, and 35 grams per liter total alkali

Changes in titratable sulphide and concentration of active alkali with the preheating are as follows:

Cooking time, min.	0	10	20	30	40	50	60
Steam pressure, p.s.i.g.	0	20	40	60	80	100	115
Liquor concentration, g. active alkali per l.	35.5	31.6	29.2	24.4	22.5	18.4	16.4
Sulphidity, %	18.0	16.6	27.7	24.5	39.9	37.9	48.4

The sulphide had dropped from the calculated value of 35 to 18% before the cook started. Titratable sulphide increased with decrease in alkali which is to be expected. More sulphide is hydrolyzed to caustic and bi-

Table V. Effect of Black Liquor Additions to Cooking Liquor Before Pulping

(Max. cooking pressure 115 p.s.i.g., 1.0 hr. to max. pressure throughout)

Black liquor in cooking liquor, %	*Active alkali, % Na_2O to wood*	*Liquor concentration, g. Na_2O/l.*	*Sulphidity, % Calculated*	*Sulphidity, % Actual*	*Total cooking time, hr.*	*Total yield, % of original wood*	*Screenings, % of total yield*	*Permanganate no.*	*Bleach requirement, % available chlorine*	*Chemical analysis of screened pulp, % Lignin*	*Alpha*	*Pentosan*
50.0	16	36.3	25.0	14.2	0	...	...	...	...	27.0	...	...
50.0	16	...	...	...	1.0	55.3	34.8	...	...	...	...	...
50.0	16	...	...	...	1.5	49.7	9.0	...	...	...	...	...
50.0	16	...	...	...	2.0	48.5	5.4	...	...	...	...	...
50.0	16	...	...	...	2.5	48.6	3.4	13.4	7.4	2.3	78.3	21.4
50.0	16	...	...	...	3.0	47.3	2.0	...	...	...	...	...
50.0	16	...	...	...	3.5	46.9	1.4	12.7	7.5	2.3	81.6	20.8
50.0	16	35.5	30.0	12.9	0	...	...	...	...	...	...	...
50.0	16	...	...	...	1.0	56.5	29.2	...	...	...	...	...
50.0	16	...	...	...	1.5	51.3	8.9	...	...	...	...	...
50.0	16	...	...	...	3.5	46.9	1.5	13.0	7.6	2.4	81.3	23.0
50.0	16	36.2	40.0	27.3	0	...	...	...	...	...	...	...
50.0	16	...	...	...	1.0	58.2	31.0	...	...	...	...	...
50.0	16	...	...	...	1.5	50.0	9.2	...	...	...	...	...
50.0	16	...	...	...	3.5	46.9	1.0	...	8.5	2.5	82.4	21.0
66.6	16	37.7	25.0	10.0	0	...	...	...	...	...	...	...
66.6	16	14.1	...	17.1	1.0	56.9	47.4	...	...	...	...	...
66.6	16	10.1	...	20.0	1.5	49.8	9.8	...	...	...	...	...
66.6	16	6.2	...	32.0	3.5	47.0	1.3	...	11.0+	2.4	83.6	20.7
66.6	16	34.4	30.0	12.2	0	...	...	...	...	...	...	...
66.6	16	12.0	...	8.8	1.0	57.8	46.6	...	...	...	...	...
66.6	16	...	...	...	1.5	50.4	12.5	...	...	...	...	...
66.6	16	5.7	...	29.5	3.5	47.2	2.8	14.1	8.7	2.1	85.8	21.2
66.6	16	35.9	40.0	23.2	0	...	...	...	...	...	...	...
66.6	16	...	...	...	1.0	56.8	28.8	...	...	...	...	...
66.6	16	...	...	...	1.5	50.0	9.3	...	...	...	...	...
66.6	16	...	...	...	3.5	46.9	1.5	...	8.3	2.7	82.2	20.6

Table VI. Effect of Black Liquor Additions on Cooking Liquor Analyses

Time between make-up and analysis, hr,	Calculated liquor analysis: Active alkali, g. Na_2O/l.	Sulphidity, %	Black liquor, %	Actual liquor analysis TAPPI Std. T 625 m-44[a]: Active alkali, g. Na_2O/l.	Sulphidity, %
1/2	35.0	35.0	0	35.6	33.9
1	35.0	35.0	25.0	35.3	22.0
1	35.0	35.0	33.3	34.9	20.3
1	35.0	35.0	66.6	36.8	17.3
19	35.0	25.0	50.0	36.3	14.2
19	35.0	30.0	50.0	35.5	12.9
19	35.0	30.0	66.6	34.4	12.2
19	35.0	40.0	50.0	36.2	27.3
19	35.0	40.0	66.6	35.9	23.2
24	35.0	35.0	25.0	35.2	23.3
24	35.0	35.0	33.3	34.4	20.3
24	35.0	35.0	66.6	35.5	9.4
43	35.0	25.0	50.0	33.8	7.7

Note: As a check of original liquor analysis, liquor analyzed by TAPPI Std. T 624 m-44, 35.3 g. Na_2O/l. active alkali, and 35.0% sulphidity (phenolphthalein and methyl orange end points).
[a] Active alkali (NaOH and Na_2S). Na_2S determined by H_2S generation.

sulphide as alkali is consumed, and, as this caustic is consumed, the sulphide becomes a higher percentage of the remaining active alkali. Comparison of this cook to the corresponding cook in which no black liquor was used (cook no. 12-35-2, Table II) showed the sulphidity at 1.5 hours to be 51.9%. The sulphidity at 1.0 hour, not shown in Table II, was 47.2%, in very close agreement with the value of 48.4 listed above. The active alkali in this white liquor cook was 10.8 grams per liter at the end of 1.0 hour as compared to 16.4 when black liquor was used. Volume of cooking liquor in both cases was 6.86 liters.

Some very interesting figures result from the above data. Total weight of sulphide, expressed as Na_2O, charged in the white liquor cook, was $6.86 \times 35.1 \times 0.342 = 82.2$ grams; for the black liquor cook the weight of sulphide charged was $6.86 \times 35.3 \times 0.35 = 84.7$ grams (calculated), of which $6.86 \times 35.3 \times 0.18 = 43.5$ grams was accounted for by titration. Therefore, the apparent disappearance of sulphide due to black liquor dilution was $84.7 - 43.5 = 41.2$ grams. At the end of 1-hour cooking time the sulphides in the cooking liquors were: for white liquor, $6.86 \times 10.8 \times 0.472 = 34.9$ grams; for the black liquor cook, $6.86 \times 16.4 \times 0.484 = 54.4$ grams. These values were also determined by titration and sum as follows:

	Grams sulphide (as Na_2O) available in cooking liquor: At start	At 1.0 hour's cooking time
100% white liquor	82.2	34.9
50% black liquor in white liquor	43.5	54.4

There is an apparent paradox in that the black liquor seems to make part of the sulphide unavailable for reaction without an appreciable lowering of active alkali. As pulping progresses and active alkali is consumed, per cent sulphidity seems to return to a value equal to that attained in a cook in which no black liquor was used. However, active alkali is not consumed as rapidly in the black liquor cook, resulting, on a percentage basis, in more sulphide than when no initial sulphide had disappeared.

It is planned to further investigate the mixing of black liquor to the cooking liquor before pulping. It may be possible that the age of the black liquor used 3 to 4 weeks, had some effect on the data reported above. More data on chemical analysis of liquors and pulps, bleach requirements and effects on yield, screenings and physical strength should be obtained before the effect of black liquor dilution can be fully evaluated.

CONCLUSIONS

From the above discussion the following conclusions may be drawn:

1. The best range of sulphidity in cooking liquors for the pulping of scrub oak is 30 to 40%. Within this range there is little difference in all-round properties of the pulp.

2. Sixteen per cent chemical (grams Na_2O per 100 grams dry wood) should be used for best results. Eighteen per cent chemical may be used as long as steam pressure does not exceed 115 p.s.i.g., but the additional chemical is in excess.

3. Concentrations of 35 grams per liter (expressed as Na_2O) will produce the best pulps. Concentrations of 20 grams per liter result in very slightly stronger pulps but require more bleach. Concentrations above 35 grams per liter are somewhat easier to bleach but their strengths are definitely inferior.

4. A cooking schedule of 1.0 hour to a maximum steam pressure of 115 p.s.i.g., with 1.5 to 2.0 hours at maximum pressure should be used. Shorter times result in stronger pulps, higher yields, and higher bleach requirements. Longer cooking times decrease yield and strength and reduce bleach requirements.

5. The above cooking schedule applies to mill-size chips (-1-inch, $+ 1/2$-inch screen openings). Smaller chips ($-1/2$-inch, $+ 1/4$-inch screen size) can be pulped to the same yield in 30 to 60 minutes less cooking time with a slight reduction in tear and burst factors.

6. Pulps produced under the conditions specified above can be classified as "easy bleaching." Bleach requirements, in single stage, are in the range of 7.0 to 8.0%. If bleached in multistage the pulps should require 5.0 to 5.6% bleach to attain a G.E. brightness of 75.

7. There appears to be no straight-line relationship between permanganate number and bleach requirements of pulps. A plot of permanganate number versus bleach requirement shows random distribution between values of permanganate numbers of 11.7 to 23.7 and bleach requirements of 6.1 to 26.0%. This lack of correlation is due only in part to changes in the pulping variables over the range of data presented.

8. Additions of black liquor to the cooking liquor before pulping cause a reduction in titratable sulphide in the cooking liquor. This sulphide disappearance increases with percentage of black liquor addition and time between mixing and analysis. The pulping reaction is milder than when no black liquor is used and screenings in the pulp are higher. More study of this problem is needed before definite conclusions may be drawn.

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Repulping Kraft Rejects

K. G. CHESLEY, P. L. GILMONT, and J. E. PARNELL

Experimental studies on repulping kraft rejects, both alone and mixed with chips, show that both the knotter rejects and the screen rejects can be repulped with chips without excessive degradation of fiber or loss in yield. The flow sheet of the Crossett paper mill repulping system is described.

In the kraft pulping industry, chip size and cooking conditions vary widely. Each mill attempts to adjust these variables to produce an optimum yield and quality of pulp. Since the optimum yield and quality of pulp will vary from mill to mill depending of the products manufactured, a great deal of difference in process variables is to be expected. One important factor which is always taken into account in selecting optimum operating conditions is the amount of incompletely pulped chips which is separated from the acceptable pulp by screening. Yields of pulp are normally expressed as "per cent screened yield" and "per cent rejects."

The material separated from acceptable pulp by the knotting and screening operation, referred to as knotter rejects and screen rejects, may be best classified by size. The larger fragments rejected by the knotters consist of partly cooked knots, large chips, and slivers. The smaller size rejects, separated in the final screening operations, consist of slivers, fiber bundles, and good stock clinging to the rejects. Bark and extraneous dirt may be found in both size classifications.

The volume and character of rejects depend on the raw material, the degree and uniformity of cooking, the extent of screening, and probably a number of other factors that are difficult to define. Very soft pulps usually contain a low percentage of rejects, even though finer screens are used. Coarse pulps may contain a high percentage of rejects. In high yield and semichemical processes, the rejects would vary from 30 to almost 100% if these pulps were screened in the same manner as the finer pulps. Usually, the refining and screening steps in these processes are modified in such a way as to eliminate most of the rejects. Rejects in pulp cooked for high strength unbleached paper usually run 3 to 10% of the pulp, approximately half knots and half screenings.

Complete delignification is neither necessary nor desirable in cooking unbleached pulps. There are definite advantages to milder cooking to conserve yield and strength, especially with respect to that fraction of the chips that is pulped most easily. However, as delignification is limited more and more, the loss in screened yield caused by excessive rejects rises sharply. Under the optimum conditions for minimum delignification and maximum screened yield the cooking becomes very sensitive to small variations. A charge slightly undercooked may contain an excessive percentage of rejects. Therefore, pulp is usually overcooked to allow a margin of safety.

Disposal or utilization of rejects depends on the local situation at each mill. The simplest disposition is waste to the sewer. This method, now being practiced less and less, wastes raw material of considerable value, increases the pollution load in mill effluent, and encourages overcooking to minimize both the waste and the pollution.

Mechanical refining of rejects is practiced in many mills where a use for the coarse fiber exists. Where a mill produces board or corrugating medium, refined screen rejects may be a very desirable addition to the furnish. Although knots can be refined they produce a coarser pulp and there is loss of salt cake which can be avoided if the knots are recooked.

Another method of disposing of rejects is to recook them in standard digesters or chemipulpers, either alone or with fresh chips. The pulp obtained by recooking would not be as coarse as that obtained by mechanical refining and should be suitable for high-strength papers. Recooking is most attractive to those mills that have no outlet for coarse fiber.

Since rejects have already been cooked once and since the screen rejects are small in size, recooking with fresh chips or under conditions required to pulp fresh wood might be expected to overcook, degrade, and destroy the fiber. Yields and strengths would be low if this were true. On the other hand, the first cook may have extracted much of the ligneous and easily decomposed carbohydrate material leaving only the more resistant cellulose. In this case, yields would be high but strengths might be reduced by degradation of cellulose and hemicellulose.

The purpose of this work was to study the effect on yield and strength of recooking rejects with and without addition of fresh chips. Since the Crossett paper mill has started to recook rejects this year, we have also the initial mill scale experience on this subject.

EXPERIMENTAL

A small vertical digester with a normal capacity of 4000 grams of chips (oven dry) was used for the experimental cooking studies. The cooking liquor was heated indirectly in a heat exchanger and circulated through the digester. On completion of cooking, the pulp was blown into a small cyclone from which it dropped into a wire-bottomed box for washing. It was then screened through a single plate, 0.010-inch cut, flat screen. After screening, the pulp was centrifuged to produce a pulp of uniform moisture content for weighing and testing according to TAPPI methods.

Chips and knotter and screen rejects were obtained from the pulp mill. The rejects are flushed from the flat screen with a stream of water giving a high water to reject ratio. However, to facilitate handling, the

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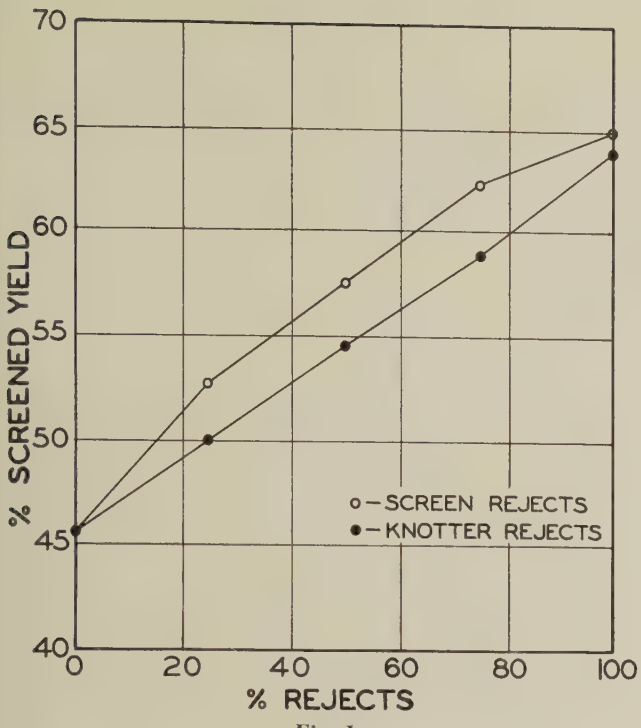

Fig. 1

rejects were squeezed out by hand to a consistency of 13 to 20% moisture-free. This consistency still allowed water to drain from top to bottom in a container of rejects so that in order to obtain a material that would remain reasonably uniform in moisture content throughout, the rejects were further dewatered in a centrifuge to approximately 30% moisture-free consistency. This left such an absorbent material that a reasonably high liquor to wood ratio was required in the digester to permit circulation. Mill white liquor was used as a base for the cooking liquor, chemical being added as required to make up the desired composition. Black liquor, adjusted to a definite density, was used for dilution to simulate mill cooking conditions.

The normal charge of either chips, chips and rejects, or rejects alone was 4000 grams on a dry basis. A liquor to wood ratio of 4.5:1 was used.

To determine the effect of cooking mixtures of chips and rejects under standard cooking conditions, a series of digester charges was made up containing 0, 25, 50, 75, and 100% knots, and another series of charges was made up with similar proportions of screen rejects. Each of these charges was cooked under the following conditions:

Total alkali to wood ratio	23%
Active alkali to wood ratio	19%
Sulphidity	19% (total chemical basis)
Total volume	18 liters
Time to 100 p.s.i.g. pressure	90 minutes
Time at pressure	45 minutes
Relief	400 ml. condensate

The most striking feature of the results of these cooks was the increase in yield on a weight basis when rejects were mixed with the chips. This is shown in Table I and Fig. 1.

The quality of the pulp decreased as the percentage of rejects increased. The beater curves (Figs. 2 and 3) show this clearly. The decrease in strength is not serious unless a sizable fraction of the furnish is derived

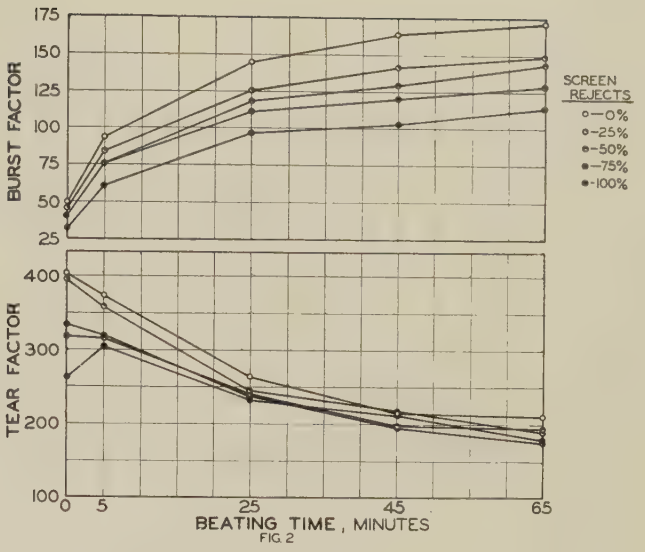

Fig. 2

from rejects. In amounts of 10% or less of the furnish, it is unlikely that the decrease in strength could be detected by the methods of pulp evaluation used in this study.

Table I. Yield Data from Experimental Cooks

Composition of digester charge, %	*Permanganate No. (TAPPI)*	*Screened pulp yield, %*	*Rejects yield, %*
100% Chips	32.8	47.6	1.6
25% Screen rejects	32.1	53.7	1.0
50% Screen rejects	30.7	57.5	0.5
75% Screen rejects	30.0	62.3	0.3
100% Screen rejects	30.2	65.3	0.3
25% Knot rejects	32.8	49.8	2.7
50% Knot rejects	32.3	54.6	2.3
75% Knot rejects	33.2	58.9	4.7
100% Knot rejects	29.7	62.7	2.9

It was noted during this study that rejects occupied a greater volume in the experimental digester than an equivalent moisture-free weight of chips. For the screen rejects alone this increase in volume amounted to 150% when the rejects were dumped in without packing at 30% moisture-free consistency. It is not known what this volume-weight relationship would be in a large digester where the rejects would be loaded with chips and cooking liquor.

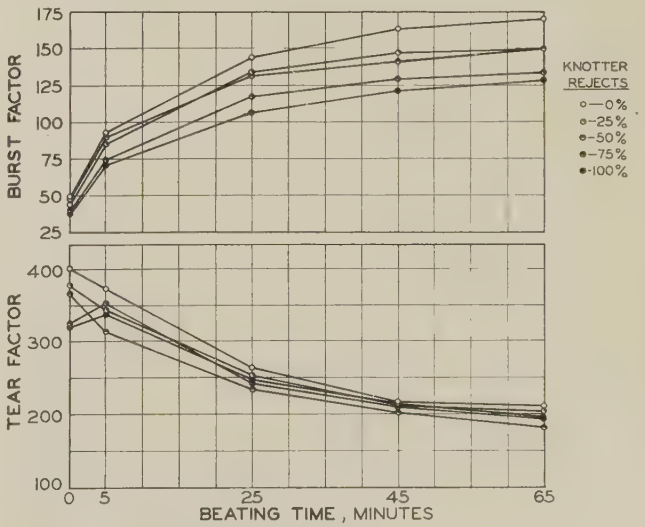

Fig. 3

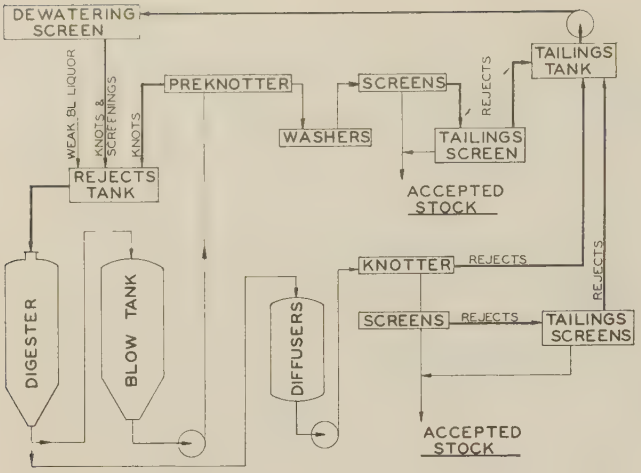

Fig. 4

Since the screen rejects have been partially pulped, it seems reasonable to assume that they could be repulped with a much less drastic treatment than that used for chips. Some experimental cooks were made with a reduced time schedule and a reduced alkali to wood ratio on the screen rejects alone. The results of this work indicated that these rejects could be pulped with either less alkali or in less time than is required for chips.

Knotter and screen rejects from a normal kraft cook, when repulped with fresh chips, give a screened yield approximately 65 to 70% of their moisture-free weight.

Pulp from recooked rejects does not have as high strength values as pulp from fresh chips. However, the yields and strength obtained show that recooking does not degrade the fiber as much as might be expected.

RECOOKING REJECTS AT CROSSETT PAPER MILL

With the installation of new countercurrent vacuum washers early this year, the Crossett paper mill made provision for recovery of rejects that had heretofore been discarded to the sewer. The rejects from the preknotters are returned directly to the reject tank effecting not only an increase in pulp yield but also a saving in salt cake and organic material that had previously gone to the sewer with the knots. The screen rejects are pumped back to the digester floor, de-watered, and dropped into the reject tank. Figure 4 shows a flow sheet of the reject recovery system.

The rejects are returned to the digesters when the weak black liquor is pumped in early in the loading procedure. This operation is probably the simplest and most economical way to transport the material back to the digesters but it introduces some difficulties.

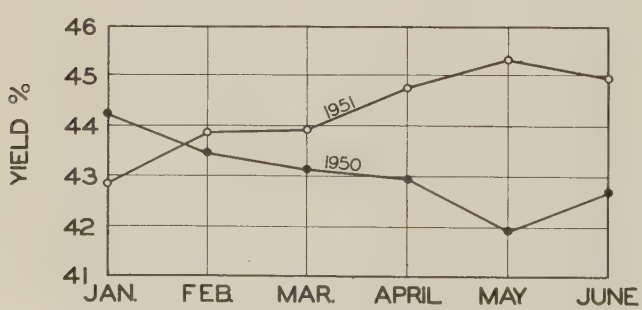

Fig. 5. Pulp mill yield

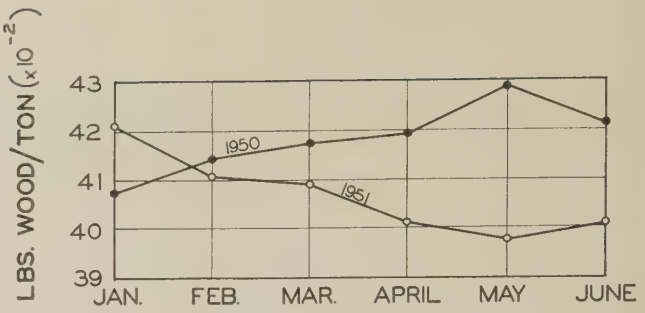

Fig. 6. Wood consumption

Since the rejects occupy volume in the tank the measurement of black liquor is not as accurate as it would otherwise be. In spite of the fact that the rejects are kept in suspension by an agitator some clogging of the pump does occur especially when the reject load is heavy. The system is unduly sensitive to variations in the volume of rejects and is easily overloaded.

Since the black liquor is always loaded first, the rejects are in the bottom of the digester where the weight of the chip charge can compress them to a minimum volume. For this reason, the digester capacity has not been adversely affected to an appreciable extent by adding rejects although the high specific volume should result in less weight being loaded. The increase in yield noted in the experimental cooks would also be a compensating factor. The yield of pulp per unit weight of wood increased almost as soon as the new installation started to operate. This increase is shown in Figs. 5 and 6. Since rejects represent less than 10% of the digester charge no perceptible loss of pulp strength was expected and none was found.

The technique of cooking is somewhat more difficult when rejects are mixed with chips. This is particularly true of the screen rejects and this is aggravated further when the rejects contain fiber stock. Circulation in the digester is adversely affected by heavy reject loads, especially when direct steaming is used. Gas relief is more difficult. There is a tendency for screens to get choked with stock, and for turpentine recovery to be reduced. Even so, the value of the pulp recovered far outweighs all the disadvantages thus far experienced.

The possibility always exists of recooking rejects separately from fresh wood or of repulping by mechanical refining. Separate recooking might be justified if the amount of rejects were sufficiently high or the efficiency of cooking rejects separately could compensate for the added expense. Mechanical defibering seems to be even more promising if future developments produce a suitable fiber for the desired grades. In either case, efficient recovery of rejects will relieve the basic pulping process of limitations now imposed by the need to maintain rejects low.

The Crossett paper mill's system of repulping rejects is not necessarily the most efficient or desirable method of recovering usable fiber from both knotter and screen rejects. It is a simple and economical installation, and should be regarded as a step forward rather than as a final solution to the problem of raw materials utilization and conservation.

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The Sorption and Rate of Sorption of the Amylose Fraction of Starch by Papermaking Fibers

WESLEY L. PEARL

The amount of amylose sorbed by papermaking fibers under varying conditions has been determined. The change in the concentration of the amylose in the solution was measured by an iodine-photocolorimetric method and an anthrone-sulphuric acid reagent method. The rate of amylose sorption by cellulose fibers was initially high, but fell off and approached zero with the sorption continuing to zero concentration without evidence of reaching an equilibrium. When the amylose concentration level was maintained, the rate of sorption decreased rapidly at first and then appeared to approach a constant value. The rate increased with a decrease in temperature. Once sorbed, the amylose apparently was not removed by a change in concentration and only partially removed by an increase in temperature. The mechanism of sorption was explained as a dual process, in which the amylose was sorbed by the cellulose surface and this amylose sorbed additional amylose. At a high solution pH, the rate of sorption was definitely retarded as compared with a low pH solution. There was a small increase in the rate of sorption with a decrease in the temperature at the high pH but of less magnitude than the increase obtained under more acid conditions. The rate of sorption for cotton linters and alpha pulps increased with the degree of beating of the pulp. Sorption of the amylopectin fraction of starch by cotton linters was established. The bursting strength was observed to increase with the amount of amylose sorbed.

STARCH has been employed for many years as a beater additive for producing certain desirable characteristics in various grades of paper. Starch may be added for one or all of the following reasons: to increase bursting and tensile strengths or folding endurance; to increase stiffness, abrasion resistance, or rattle; to improve sizing or finish; or to lay surface fuzz. Despite this general acceptance of starch as a valuable part of the furnish, the principles underlying its usage are not well understood. Particularly in question is the mechanism of retention of starch by papermaking fibers. Some maintain that starch is flocculated onto the fiber by alum and held by an "electrostatic cement" (*1, 2*). Other investigators (*3, 4*) claim that starch is flocked out by rosin and alum and merely filtered by the pulp on the wire; still others (*5, 6*) favor a coflocculation theory.

Much of the misunderstanding of the role of starch in papermaking can be traced to the incomplete knowledge of its chemistry and structure. It has only been since about 1940 that the existence of more than one kind of molecule in starch has been definitely established. As a result of improved methods of fractionating, two components of starch with quite different structural patterns have been isolated: amylose, a straight-chain polymer and amylopectin, in which the regularities of the linear chain are interrupted by frequent branching. A number of reviews on the development of the theory of the structure of starch have appeared recently (*7–10*).

The straight chain or amylose fraction of starch has been reported to be selectively adsorbed from starch solutions by cellulose fibers (*11–13*). The principal objective of this study was to characterize the mechanism of this retention of the amylose fraction of starch by papermaking fibers. Particular emphasis was placed on the rate of sorption of the amylose from solution. Some experiments were also made to compare the retention of the amylose and the amylopectin fractions.

PREPARATION OF MATERIALS

Raw Materials

Corn Products Refining Co. Globe brand pearl starch number 144 was used as the raw material for fractionation. Hercules' cotton linters, Weyerhaeuser's bleached sulphite, and Rayaceta alpha pulps were used. The pulps were beaten to the desired Schopper-Riegler freenesses in a Valley laboratory beater according to TAPPI Standard T 200 m-45.

Fractionation of Cornstarch

Primary separation was effected by selective precipitation of the linear amylose component with Pentasol according to the method of Lansky, Kooi, and Schoch (*14*) in which they boiled the starch under reflux instead of autoclaving. The linear component was purified by three recrystallizations from butyl alcohol. The original supernatant solution from the supercentrifuge was refrigerated for 2 weeks, supercentrifuged again (using a fine orifice of 0.0430 inch diameter into the rotor), and the amylopectin was isolated in the usual manner (*14*). The iodine-binding capacity of the fractions was measured potentiometrically by the method of Bates, French, and Rundle (*15*) as modified by Lansky, et al. (*14*). These iodine-affinity values for different batches were found to vary from 16.2 to 20.1 for amylose and from 0.8 to 1.2 for amylopectin.

Preparation of Solutions of Known Concentration

The starch or starch fractions were screened through a 40-mesh wire, introduced into a beaker of mechanically agitated boiling water, and stirred for 20 minutes. Enough starch or amylopectin was added to give a solution containing 3 to 5 grams per liter; the concentration of the amylose was 1 to 2 grams per liter. The

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exact concentrations of the solutions were determined by means of Dreywood's (*16*) color reaction of carbohydrates with anthrone in sulphuric acid in a manner similar to that used by Shriver, Webb, and Swanson (*17*). A known weight of glucose was used for calibrating the anthrone reagent [a similar glucose-calibration technique was subsequently described by McCready, et al. (*18*)]. The glucose concentrations were multiplied by 0.90 to convert to the equivalent starch concentration and to establish a log transmittance versus concentration calibration plot for use with the starch materials.

METHODS FOR RETENTION BY SORPTION

Sample Preparation

Pulp samples [usually 1.2 grams (oven-dry basis)] of known moisture content were weighed with an accuracy of 1 mg. and placed in an 8-ounce narrow-mouth bottle (cover equipped with a rubber gasket). A measured volume of distilled water (ordinarily 100 ml.) was introduced into the bottle and the pulp was carefully dispersed by means of a mechanical stirrer. Sufficient 0.01 *N* hydrochloric acid was added to give a final pH value of 4. The necessary amount of starch or starch-fraction solution was then added to give the required final concentration. The final volume was brought to 200 ml. with distilled water. The sorption bottles were then rotated in a constant temperature bath at the temperature and for the time desired.

Amylose solutions at a pH of 4 or lower were stable for times up to 500 hours, whereas retrogradation occurred in much shorter times at pH values between 4.5 and 7. No distinct difference was noted between the amount sorbed at pH values of 4 and 7 up to 45 hours, other conditions being equal.

Analytical Procedures for Determination of the Amount Sorbed

Two analytical methods were used; both are based on the measurement of the change in concentration of the solute in the solvent.

Iodine-Photocolorimetric Method. The photocolorimetric determination of starch by means of the complex formed with iodine [Simerl and Browning (*19*)] was modified for the present purpose. The sample bottle was removed from the rotator and allowed to stand for 15 minutes at the same temperature as rotated. A 5-ml. sample of supernatant liquor was pipetted from the settled pulp suspension (centrifuged or filtered through a coarse fritted funnel if necessary), placed in a volumetric flask, and diluted to the proper volume. The desirable range of transmittance for colorimetric work is 20 to 50%, with 6 to 70% transmittance allowable (*20*). Whole starch solutions were diluted to a concentration of 0.01 to 0.05 gram per liter, amylose solutions were diluted to 0.005 to 0.03 gram per liter, and amylopectin solutions were diluted to 0.05 to 0.25 gram per liter to obtain the proper transmittance range when using a 1-cm. absorption cell.

Ten milliliters of the diluted solution were pipetted into a test tube, to which was added the necessary amount of standard iodine solution (1.0 gram of iodine and 1.5 grams of potassium iodide per liter) to assure complete reaction with the starch material. A minimum iodine-starch ratio of 1:2, iodine-amylose ratio of 1:1, or iodine-amylopectin ratio of 1:1 was required. One milliliter of iodine solution was used for whole starch and amylopectin solutions and 0.3 ml. for amylose solutions. The mixture was well shaken and poured into a 1.0-cm. absorption cell. The transmittance was determined with a Coleman Model 14 Universal Spectrophotometer using a reference cell containing the same dilution of the standard iodine solution in distilled water. Percentage transmittance readings were taken at wavelengths of 625 mmu for amylose and starch solutions and 570 mmu for amylopectin solutions. The concentration of starch material present in the original liquor was determined from a calibration curve that was obtained by testing 5 ml. of the original starch or starch-fraction solution (exact concentration determined with anthrone and glucose) in a similar manner.

Anthrone-Reagent Method. After complete removal of all cellulosic material from solution, the anthrone-sulphuric acid reagent method (*16*) as modified by Shriver, et al. (*17*) was used. The following procedure removed all cellulosic material present. Fifty milliliters of the pulp slurry from the sample bottle were poured rapidly into a coarse fritted funnel mounted in a 125-ml. filter flask attached to a vacuum line. When the pulp pad in the funnel was sucked nearly dry, a clean filter flask was placed in the line and another 20 ml. of slurry were poured into the fritted funnel. Fifteen milliliters of the solution which had passed through the pulp pad were poured into a 15-ml. centrifuge tube containing a glass-wool plug at the bottom to entrap any stray cellulosic material present. The solution was centrifuged for a minimum of 15 minutes at full speed in an International Clinical Centrifuge. Five milliliters of the centrifuged solution were diluted until the concentration of carbohydrate was between 0.02 and 0.04 gram per liter. Another 5 ml. of the centrifuged solution was analyzed by the iodine-photocolorimetric method for comparison.

The iodine-photocolorimetric method is based on the unique complex formed between iodine and starchy materials, is affected by variation in chain length of the amylose molecules (*21*), and is subject to the configuration of the starch substances (*15*, *21*, *22*). The anthrone-reagent method measures the total carbohydrate content and is not dependent on starch variations, but is highly sensitive to the presence of cellulose residues. The iodine-photocolorimetric method gave the highest degree of precision, was most sensitive in measuring small changes in concentrations and low concentrations and, therefore, was used extensively in the present work.

The effect of the variation in iodine affinity values of the different batches of the amylose fraction was tested by making identical sorption tests concurrently with amylose solutions prepared from three different batches ranging in iodine affinity from 16 to 20. No significant difference was noted in the amount of amylose sorbed by cellulose when an amylose from different batches was used.

RATE OF SORPTION

In the development of an analytical procedure for determining the starchy material sorbed on pulp, the time to reach sorption equilibrium was desired. Several experiments were run at 25.0°C. Analyses were

made by the iodine-photocolorimetric method on samples of the supernatant liquor. When several aliquots were obtained from one experiment, allowance was made for the amylose removed. The results of the experiments are given in Table I and are plotted in Fig. 1. The system is characterized by an initially high rate of sorption, which gradually decreases with time. Sorption continues until all the amylose in solution has been sorbed. The number of grams of amylose that would be sorbed per 100 grams of pulp (x/m) when all the amylose has been removed from solution is listed in Fig. 1.

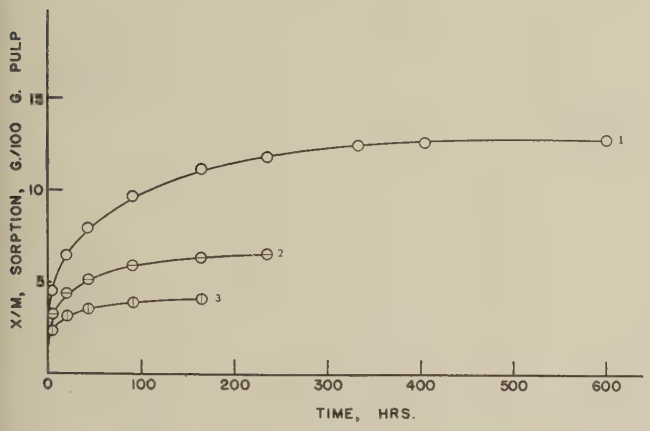

Fig. 1. Rate of sorption of amylose

	Initial concentration, g./l.	x/m when complete, g./100 g. pulp
(1)	0.824	12.95
(2)	0.412	6.63
(3)	0.247	4.07

25.0°C.

Similar runs were made at 4.2 and 37.6°C.; the data are also tabulated in Table I. Measurements were made until the concentration of the amylose in the solution was too low to determine. Again, the sorption rate decreased with time and no sorption equilibrium was reached short of exhaustion of the amylose in the solution. The curves for the different temperatures at equal initial concentrations are compared in Fig. 2. The initial sorption rate increases with a decrease in temperature.

A similar experiment was carried out using fresh samples instead of continually sampling from the same specimens. This run corroborated the falling rate and exhaustion tendencies previously obtained.

A test was made to check the dependence of the sorption rate on the amount already sorbed. One sample (sample A, Table II) was prepared with 75 ml. of an amylose solution containing 1.30 grams per liter (to give 200 ml. of a solution containing 0.488 gram per liter) and allowed to sorb at 4.2°C. for 72 hours. The concentration, determined at various times, is given in Table II. A second sample (sample B, Table II) was prepared in a similar manner and allowed to sorb for 24 hours at 4.2°C. The sample was tested and another 75 ml. of the amylose solution (1.30 grams per liter) were added and sorption was carried out another 24 hours. Again the sample was tested and treated with 75 ml. of the above amylose solution. The concentration of sample B was also determined after 72 and 96 hours. The results are listed in Table II. The data show that the rate of sorption is primarily a function of the concentration of the amylose present in the solution for each time period and slightly influenced by the amount already sorbed. The repeated addition of

Table I. Rate of Sorption of Amylose by Cotton Linters
(400-ml. Schopper-Riegler Freeness)
Iodine-Photocolorimetric Method

Temp., °C.	*Initial concn., g./l.*	*Time, hr.*	*Total volume, ml.*	*Instantaneous concn., g./l.*	*Indicated sorption, g./100 g. pulp*
25.0	0.824	5.2	200	0.550	4.56
		20	195	0.436	6.45
		43	190	0.342	7.94
		91	185	0.233	9.62
		164	180	0.134	11.12
		235	175	0.086	11.80
		332	170	0.038	12.48
		404	165	0.025	12.67
		600	155	0.006	12.92
	0.412	5.2	200	0.218	3.23
		20	195	0.146	4.40
		43	190	0.098	5.17
		91	185	0.052	5.88
		164	180	0.020	6.35
		235	170	0.005	6.57
	0.247	5.2	200	0.106	2.35
		20	195	0.058	3.13
		43	190	0.031	3.56
		91	185	0.010	3.87
		164	180	0.0	4.03
37.6	0.824	5.3	200	0.620	3.40
		20	195	0.532	4.83
		43	190	0.475	5.73
		91	185	0.346	7.72
		164	180	0.252	9.14
		235	175	0.201	9.88
		332	170	0.161	10.43
		404	165	0.135	10.80
		600	160	0.086	11.43
	0.412	5.3	200	0.268	2.40
		20	195	0.206	3.40
		43	190	0.167	4.02
		91	185	0.107	4.96
		164	180	0.061	5.65
		235	175	0.038	5.98
		332	170	0.019	6.26
		404	160	0.011	6.36
		600	150	0.002	6.48
	0.124	5.3	200	0.050	1.23
		20	195	0.021	1.70
		43	190	0.012	1.85
		91	185	0.0	2.45
4.2	1.030	5.8	200	0.550	8.00
		20	195	0.324	11.70
		43	190	0.214	13.43
		91	185	0.127	14.80
		164	180	0.074	15.58
		235	175	0.047	15.89
		332	170	0.024	16.30
		404	165	0.013	16.46
		600	155	0.003	16.58
	0.824	5.8	200	0.404	7.00
		20	195	0.226	9.89
		43	190	0.146	11.15
		91	185	0.083	12.12
		164	180	0.046	12.70
		235	175	0.027	12.97
		332	165	0.015	13.15
	0.618	5.8	200	0.262	5.93
		20	195	0.126	8.14
		43	190	0.067	9.09
		91	185	0.029	9.67
		164	180	0.011	9.94
	0.412	5.8	200	0.136	4.60
		20	195	0.044	6.10
		43	190	0.017	6.52
		91	185	0.004	6.73
	0.247	5.8	200	0.047	3.34
		20	195	0.007	3.98
		43	190	0.0	4.10

amylose to sample B caused it to continue to sorb rather than level off as did sample A. The latter leveled off toward exhaustion of the amylose in solution. From sample B, Table II, it is seen that the concentration in the 48 to 72-hour period was at no time as low as the concentration at the end of the 24 to 48-hour period, yet more was sorbed in the earlier period. This, together with other data, indicates that the rate of sorption is more rapid in the early phases than at the latter stages at equal concentrations.

Approach to an Infinite Bath

By eliminating concentration as a variable, the other factors affecting the rate of sorption could be better evaluated. This could be accomplished by use of an

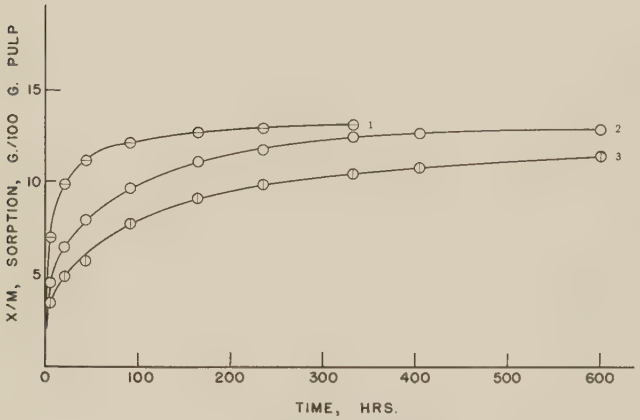

Fig. 2. Rate of sorption of amylose

	Temperature, °C.	x/m when complete, g./100 g. pulp
(1)	4.2	13.32
(2)	25.0	12.95
(3)	37.6	12.55

Initial concentration, 0.824 g./l.

infinite-bath technique—i.e., enough solution so that the concentration would not change with sorption. The analytical methods available in the present work

Table II. Effect of Change of Concentration on Rate of Sorption of Amylose

400-Ml. S.-R. Cotton Linters
Iodine-Photocolorimetric Method

Sample	*Original time, hr.*	*Initial concn., g./l.*	*Final time, hr.*	*Final concn., g./l.*	*Indicated sorption during period, g./100 g. pulp*
A	0	0.488	7.4	0.168	5.33
	7.4	0.168	24.0	0.066	1.66
	24.0	0.066	48.0	0.036	0.55
	48.0	0.036	72.0	0.023	0.20
B	0	0.488	24.0	0.066	6.99
	24.0	0.403	48.0	0.232	3.96
	48.0	0.465	72.0	0.316	3.43
	72.0	0.316	96.0	0.238	2.22

Sample A—x/m when complete is 8.04 g./100 g. pulp. 4.2°C.

(based on measurement of amount sorbed by concentration difference) were not designed for such an infinite bath-type of experiment. Such a system was approached by working at very low pulp consistencies so that concentration changes were small but measurable. Experiments were carried out in the normal manner, using 0.1 gram of pulp instead of the customary 1.2 grams of pulp in a 200-ml. volume. The temperature and initial concentrations were varied as indicated in Table III. One set of tests was made with 1.2 grams of pulp for comparison. The amount sorbed per unit of pulp at any given time is much greater for those samples

Table III. Effect of Pulp Consistency on Rate of Sorption of Amylose

585-Ml. S.-R. Cotton Linters
Iodine-Photocolorimetric Method

Temp., °C.	*Pulp, g.*	*Initial concn., g./l.*	*Time, hr.*	*Total volume, ml.*	*Instantaneous concn., g./l.*	*Indicated sorption, g./100 g. pulp*
25.0	1.2	0.490	11	200	0.276	3.56
			22	195	0.222	4.44
			44	190	0.169	5.28
			66	185	0.135	5.57
			107	180	0.082	6.36
	0.1	0.490	11	200	0.450	8.00
			22	195	0.444	9.30
			44	190	0.420	13.7
			66	185	0.402	17.0
			107	180	0.366	23.5
		0.562	2	200	0.546	3.2
			19	200	0.544	3.6
			46	195	0.525	7.3
			72	195	0.525	7.3
			96	190	0.512	9.9
			152	185	0.488	14.3
			192	185	0.476	16.5
		0.225	2	200	0.218	1.4
			19	200	0.208	3.4
			46	195	0.203	4.3
			72	195	0.197	5.6
			96	190	0.193	6.3
			152	185	0.176	9.5
			192	185	0.176	9.6
4.2	1.2	0.490	11	200	0.161	5.48
			22	200	0.108	6.35
			44	195	0.062	7.10
			66	190	0.042	7.40
			107	185	0.024	7.80
	0.1	0.490	11	200	0.444	9.20
			22	200	0.420	14.0
			44	195	0.384	20.9
			66	195	0.374	23.0
			107	190	0.338	29.7
		0.562	2	200	0.545	3.4
			19	200	0.522	8.0
			46	195	0.486	14.8
			72	195	0.472	17.8
			96	190	0.445	22.8
			152	185	0.406	30.1
			192	185	0.384	34.1
		0.225	2	200	0.215	2.0
			19	200	0.196	5.8
			46	195	0.183	8.2
			72	195	0.176	9.7
			96	190	0.170	10.9
			152	185	0.157	13.1
			192	180	0.150	14.5

containing less pulp, all other factors remaining the same. This would be expected if the rate of sorption is dependent on solution concentration, since the change in concentration is much less for samples containing the least pulp. Plots of the amount sorbed (x/m) versus time (Fig. 3 shows such a plot for data at 4.2°C.) indicated that the rate of sorption decreased only slowly after an initial rapid decrease. By dividing the amount sorbed in a small interval of time by the average con-

centration during that time, the amount sorbed at unit concentration during the time interval was obtained. By making the time intervals small enough, the average concentration was applicable in the range of small change in rate of sorption with time. Table IV lists the calculated values from some of the data in Table III. The values of $(\Delta x/m)/c_{av.}$ appear to approach a constant value in Table IV after a very rapid initial decrease. In other words, sorption is apparently approaching a constant rate when the solution concentration remains constant.

SORPTION REVERSIBILITY

Effect of Concentration

Pacsu and Mullen (*11*) and Kerr and Severson (*23*) have indicated a certain amount of irreversibility of amylose that has been sorbed by cellulosic materials.

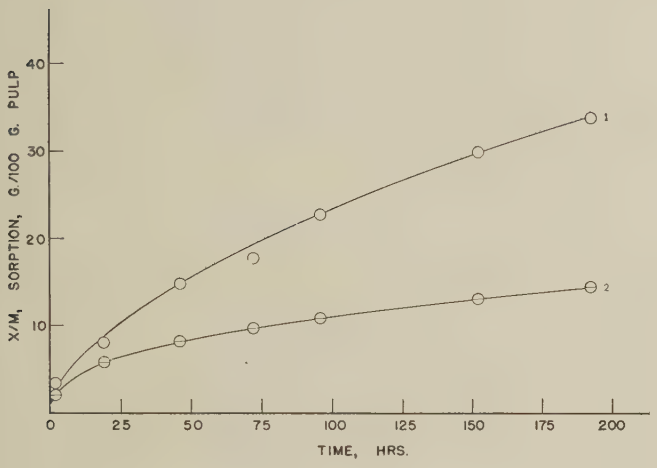

Fig. 3. Rate of sorption of amylose

(1) Initial concentration, 0.562 g./l.
(2) Initial concentration, 0.225 g./l.
0.1g. pulp; 4.2°C

The fact that sorption systems go to exhaustion is another indication of the probable irreversibility of sorption with respect to concentration. The results of an experiment to determine the effect of concentration on sorption reversibility are tabulated in Table V. Three duplicate sets of samples were set up in the usual manner; one set (samples E and F) contained one fourth the amylose concentration of the other two sets (samples C and D). The concentrations of all samples were measured after 20 hours of rotation at 25.0°C. One set (samples A and B) of the higher amylose concentration samples was diluted with 3 volumes of distilled water. The system then contained the same amylose-water ratio as the original dilute samples E and F after making allowance for the test sample removed. The three sets were then rotated another 26 hours and the concentrations were determined. The amount sorbed by the original dilute E and F samples was 40% of that of the undiluted samples (C and D). Samples A and B (containing the same amylose content as samples C and D) continued to sorb after dilution an amount only 10% less than the amount sorbed by C and D in the same time. Thus, it is concluded that the system is irreversible with regard to concentration under isothermal conditions. The lower rate of sorption of samples A and B, as compared with samples C and D for the final 26 hours, was caused by the lower solution concentration in samples A and B during the 26-hour period.

Effect of Temperature

The effect of a change in temperature on the reversibility of sorption was tested as follows. A run was made with samples at 4.2°C. for the first 24 hours. After determination of the amylose concentration at that time, two samples were held at each of the following temperatures for the next 24 hours: 4.2, 37.6, 50, 75, and 100°C. In all cases, except at 4.2°C. (see Table VI), the amount sorbed after 48 hours was less than after 24 hours. The magnitude of the sorption reversal increased with increasing temperature. Upon completion of sampling after 48 hours, some of the specimens were returned to the 4.2°C. bath for another 24 hours. The results showed that all samples returned to the 4.2°C. bath sorbed approximately the same quantity of amylose in 72 hours as did those samples which had

Table IV. Rate of Sorption of Amylose under Infinite-Bath Conditions

Data from Table III

Temp., °C.	c_0, g./l.	t, hr.	x/m, g./100 g. pulp	Δx/m, g./100 g. pulp	c, g./l.	$c_{av.}$, g./l.	$(\Delta x/m)/c_{av.}$, g./100 g. pulp
4.2	0.225	30	6.95	6.95	0.185	0.205	33.9
		60	9.20	2.25	0.173	0.179	12.6
		90	10.60	1.40	0.165	0.169	8.30
		120	11.90	1.30	0.158	0.161	8.05
		150	13.10	1.20	0.151	0.154	7.78
		180	14.15	1.05	0.145	0.148	7.10
	0.562	30	11.50	11.50	0.490	0.526	21.9
		60	17.25	5.75	0.460	0.475	12.1
		90	21.90	4.65	0.434	0.447	10.4
		120	26.00	4.10	0.412	0.423	9.70
		150	29.80	3.80	0.391	0.401	9.50
		180	33.30	3.50	0.364	0.377	9.30
25.0	0.225	30	3.78	3.78	0.200	0.212	17.8
		60	5.10	1.32	0.194	0.197	6.70
		90	6.20	1.11	0.188	0.191	5.76
		120	7.21	1.01	0.182	0.185	5.46
		150	8.21	1.00	0.176	0.179	5.59
		180	9.10	0.89	0.171	0.173	5.15
	0.562	30	4.65	4.65	0.525	0.543	8.57
		60	7.35	2.70	0.512	0.518	5.22
		90	9.71	2.36	0.487	0.499	4.73
		120	11.97	2.26	0.475	0.481	4.70
		150	14.08	2.11	0.452	0.464	4.55
		180	16.00	1.92	0.430	0.441	4.35

Table V. Effect of Concentration on Reversibility of Sorption of Amylose

400-Ml. S.-R. Cotton Linters
Iodine-Photocolorimetric Method
25°C.

Sample	Initial concn., g/l.	20 Hours indicated sorption, g./100 g. pulp	46 Hours indicated sorption, g./100 g. pulp
A[a]	0.756	5.67	6.36
B[a]	0.756	5.62	6.69
C	0.756	5.64	7.25
D	0.756	5.50	7.06
E	0.189	2.45	2.82
F	0.189	2.50	2.88

[a] Samples A and B diluted 1 to 4 after 20 hours so that the original amylose concentration ratio corresponded to that of samples E and F.

remained at 4.2°C. for 72 hours. Those samples not returned to the cold bath after 48 hours showed a slight increase in sorption for the next 24 hours.

Another run was made at 37.6°C. for the first 24 hours, at 37.6 and 4.2°C. for the second 24 hours, and at 37.6°C. from 48 to 72 hours. The increase in temperature after 24 hours resulted in a large increase in the amount sorbed and the total was about equal to that sorbed by a sample at 4.2°C. for the total time. Again a slight decrease in amount sorbed is noted when the bath temperature was raised for 24 hours, though much of the amount sorbed at the lower temperature was not given up. The above runs indicate two important facts: (1) the rate of sorption increases with decrease in temperature and (2) the amount sorbed is somewhat but not completely reversible with increase in temperature. No further work was carried out to investigate the apparent anomaly between the tendency of sorption to continue to exhaustion and the relatively large reversal of sorption at temperatures above 37.6°C.

MECHANISM OF AMYLOSE RETENTION

Several facts have been established in regard to the rate of amylose sorption by cotton linters. The rate of sorption was initially high, then fell, and approached zero with the sorption continuing to zero concentration without evidence of reaching equilibrium. When the amylose concentration level was maintained, the rate of sorption decreased rapidly at first and then appeared to approach a constant value. The rate increased with decrease in temperature. Once sorbed, the amylose was apparently not removed by a change in concentration and only partially removed by an increase in temperature.

A logical explanation of the mechanism of the sorption can be made on the basis of a dual process. The amylose would be sorbed by the cellulose surface and this amylose in turn may sorb additional amylose. The latter process may be related to the process of amylose retrogradation. The possibility of "retrogradation" sorption was demonstrated by the retrogradation of amylose from a stable amylose solution not containing pulp when the solution was seeded with retrograded amylose. The rate of this retrogradation of amylose from solution was dependent on the solution concentration. The rates of sorption of amylose by cellulose and by retrogradation would vary depending on the respective amounts of available cellulose and amylose surfaces. The rate of amylose sorption by cellulose is apparently greater than the rate of retrogradation sorption because (1) the rate of sorption was always greater at the start of the sorption than after some time had elapsed, even though a concentration level was maintained (see Tables II and IV) and because (2) the rate was greater for sorptions starting at low initial concentrations when at the same concentration level as sorption tests having higher initial concentrations (see Table I). It has been estimated that the former rate is about 3 to 10 times the latter. Comparison of the nearly constant values of $(\Delta x/m)/c_{av.}$ (Table IV) approached at 4.2 and 25.0°C. indicates that the rate of retrogradation sorption at 4.2°C. is about double the rate at 25.0°C. for equal sorbing areas.

Table VI. Effect of Temperature on Reversibility of Sorption of Amylose

1.2 G. of 400-Ml. S.-R. Cotton Linters in 200 Ml. Volume
Iodine-Photocolorimetric Method
Initial Amylose Concentration—0.488 G./L.

	0 to 24 hours			24 to 48 hours			48 to 72 hours		
Sample	*Bath temp., °C.*	*Instantaneous concn. at 24 hr., g./l.*	*Indicated sorption at 24 hr., g./100 g. pulp*	*Bath temp., °C.*	*Instantaneous concn. at 24 hr., g./l.*	*Indicated sorption at 24 hr., g./100 g. pulp*	*Bath temp., °C.*	*Instantaneous concn. at 24 hr., g./l.*	*Indicated sorption at 24 hr., g./100 g. pulp*
A	4.2	0	...	4.2	0.035	7.55	4.2	0.023	7.74
B	4.2	...	...	4.2	0.036	7.54	4.2	0.023	7.72
C[a]	37.6	0.258	3.81	4.2	0.039	7.28	37.6	0.050	7.10
D	37.6	...	...	4.2	0.044	7.40	4.2	0.023	7.74
E[a]	4.2	0.066	6.99	37.6	0.069	6.95	4.2	0.016	7.78
F	4.2	...	...	37.6	0.074	6.90	37.6	0.065	7.05
G	37.6	0.264	3.73	37.6	0.222	4.41	37.6	0.195	4.84
H	37.6	...	...	37.6	0.211	4.61	37.6	0.178	5.15
I	4.2	...	...	50	0.106	6.37	4.2	0.015	7.77
J	4.2	...	...	50	0.107	6.35	50	0.100	6.47
K	4.2	...	...	75	0.205	4.71	4.2	0.013	7.85
L	4.2	...	...	75	0.208	4.67	75	0.198	4.83
M	4.2	...	...	100	0.330	3.35	...	...	...
N	4.2	...	...	100	0.340	3.46	..	...	...

[a] Sample C at 7.4 hr.—instantaneous concentration 0.324 g./l.; indicated sorption 2.73 g./100 g. pulp. Sample E at 7.4 hr.—instantaneous concentration 0.168 g./l.; indicated sorption 5.34 g./100 g. pulp.

The actual method of attachment of the amylose to the cellulose is probably similar to the attachment of amylose to amylose because of their very similar physical properties. The attachment is probably by means of hydrogen bonding through the numerous hydroxyl groups available on amylose and cellulose. A good review of hydrogen bonding in starch and starch fractions is given by Caeser (*24*).

When Kerr and Schink (*25*) observed that a 1% pickup of amylose resulted in no increase in the bursting strength of bleached sulphite handsheets, they suggested a similar mechanism; "apparently the linear polymer fraction of cornstarch is able to orient with other linear polymer fractions such as cellulose, in much the same irreversible manner as it orients with itself so as to prevent the formation of a continuous bond."

Their observations on bursting strength will be considered later.

EFFECT OF HIGH pH ON SORPTION

The rate of amylose sorption by 400-ml. S.-R. freeness cotton linters under alkaline conditions (pH 10.4) was measured at various initial amylose solution concentrations and various temperatures. Concurrent runs were made at a pH of 4 for purposes of comparison. Figure 4 compares one such set of data for three temperatures at the same initial concentration of amylose solution. Since a very dilute solution of sodium hydroxide (5×10^{-4} N) was used to raise the pH, the sodium ions were apparently hydrolytically adsorbed by the cellulose and the liberated hydrogen ions tended to neutralize the solution over a period of time. Therefore, data were not taken after 100 hours, at which time the pH of the solution had dropped below 9. The rate of sorption is definitely retarded under these alkaline conditions as compared with experiments carried out at a pH of 4. There is a small increase in rate of sorption with decrease in temperature at the high pH but of less magnitude than the increase obtained under more acid conditions. The process of amylose retrogradation would be arrested in a dilute alkaline solution (*26*). The initial rate of sorption also has been retarded, suggesting that both amylose-by-cellulose sorption and retrogradation sorption are retarded at a high pH.

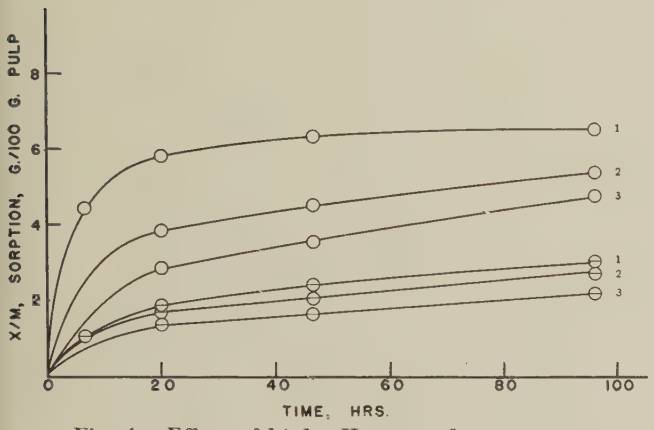

Fig. 4. Effect of high pH on amylose sorption

(1) 4.2°C.
(2) 25.0°C.
(3) 37.6°C.

Initial concentration, 0.405 g./l.
x/m when complete, 6.75 g./100 g. pulp.

Hollow circles, 4.0 pH.
Half circles, 10.4 pH.

EFFECT OF BEATING ON RATE OF SORPTION

The rate of sorption for cotton linters and alpha pulps increased with the degree of beating of the pulp. The amount sorbed at any time was higher, the higher the initial concentration; this indicates no approach to a point of saturation. This finding does not agree with Kerr's (*27*) interpretation of the results of Kerr and Severson (*23*) that cellulose fibers will adsorb up to 1% of their weight of corn amylose.

No direct relationship was found between the degree of beating of bleached sulphite pulp and the rate of amylose sorption. A resinous material located in the ray cells of the pulp was observed to be the probable cause of the apparent anomaly. When released by mechanical action, this resinous material was apparently responsible for the formation of the amylose-cellulose bond. When the ray cells were removed from the bleached sulphite by screen classification before beating, the rate of sorption increased with degree of beating.

SELECTIVE SORPTION

Because of the probable range in molecular weight of the molecules in a solution of amylose, the possibility of selective sorption was qualitatively investigated. Since the wavelength of the absorption peak of the amylose-iodine complex varies with the chain length of the amylose molecules (*21*), the accuracy of the concentration determined by the iodine-photocolorimetric method of analysis would be affected by selective sorption. The anthrone-reagent method of analysis makes no distinction between amylose chain lengths, but measures only the amount of carbohydrate present in the amylose solution. Therefore, apparent sorption indicated by the former method and not by the latter might be caused by selective sorption. Some such differences were noted with systems of low concentrations at temperatures of 25°C. and higher. The sorptions carried out under alkaline conditions revealed such a difference in indicated sorption at all temperatures. The long-chain molecules were selectively sorbed under these conditions. Generally, both methods of analysis were used when there was reason to doubt the accuracy of the iodine-photocolorimetric method.

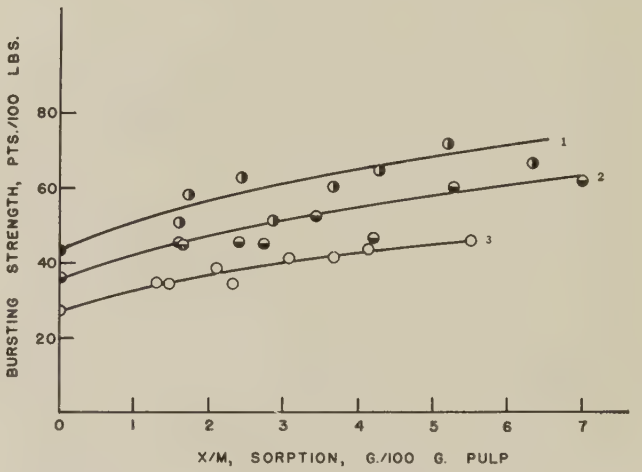

Fig. 5. Effect of amylose on bursting strength of cotton linters

(1) 400 S.-R. freeness.
(2) 585 S.-R. freeness.
(3) 770 S.-R. freeness.

COMPARISON OF THE RATES OF AMYLOSE AND AMYLOPECTIN SORPTION

In view of the findings of Tanret (*12*), Pacsu and Mullen (*11*), and Samec (*13*) that the amylose fraction of starch was selectively sorbed by cellulose, the possible sorption of amylopectin was checked. The amount of amylopectin sorbed by 400-ml. S.-R. freeness cotton linters at 3 time intervals is reported in Table VII.

The iodine-photocolorimetric and the anthrone-reagent methods of analysis were used to minimize the possible error caused by the presence of a small residue of amylose in the amylopectin fraction. The magnitude of the

Table VII. Sorption of Amylopectin by Cotton Linters

400-Ml.-S.R.Cotton Linters
Initial Amylopectin Concentration—0.287 g./l.

			Iodine-photocolorimetric method[a]		*Anthrone reagent*	
Time, hr.	*Freeness, ml. S.-R.*	*Temp., °C.*	*Instantaneous concn., g./l.*	*Indicated sorption, g./100 g. pulp*	*Instantaneous concn., g./l.*	*Indicated sorption, g./100 g. pulp*
24	400	4.2	0.117	2.83	0.118	2.82
	400	37.6	0.162	2.08	0.174	1.88
	400	25.0	0.143	2.40	0.160	2.12
	585	25.0	0.181	1.77	...	...
	770	25.0	0.204	1.38	...	...
	Unbeaten	25.0	0.251	0.60	...	...
48	400	4.2	0.086	3.35	0.091	3.27
	400	37.6	0.140	2.45	0.153	2.24
	400	25.0	0.138	2.48	0.146	2.35
	585	25.0	0.154	2.14	0.153	2.16
	770	25.0	0.180	1.70	0.177	1.75
	Unbeaten	25.0	0.242	0.64	0.245	0.59
170	400	4.2	...	...	0.020	5.26
	400	37.6	...	...	0.112	3.45
	400	25.0	...	...	0.122	3.28

[a] Run at wavelength of 570 mmu.

amount sorbed definitely establishes amylopectin sorption by cotton linters. The rate of sorption of the amylopectin fraction was much less than the corresponding rate for the amylose fraction.

STRENGTH PROPERTIES

Several of the pulps treated with amylose were made into handsheets according to TAPPI Standard T 205 m-47. The bursting strengths of various cotton linters handsheets were plotted against the respective amounts of amylose that had been sorbed (see Fig. 5). The results indicate that bursting strength increases with the amount of amylose sorbed. The strength improvement increases to a value of 60 to 75% over the original pulp for about 5% amylose retention. The results do not corroborate those of Kerr and Schink (*25*), who observed no significant effect of the sorption of 1% amylose on the resulting bursting strength.

The bursting strength for amylopectin-treated handsheets was significantly greater than that of handsheets containing an equal amount of amylose. For example, a handsheet made of 400-ml. S.-R. freeness cotton linters showed a 40% increase in strength when 0.8% amylopectin had been sorbed and a 20% increase when the same amount of amylose had been sorbed.

PRACTICAL IMPLICATIONS

Although the study of the role of starch and starch fractions in papermaking has been only started some experimental results may have practical applications worthy of note.

Experimental evidence is now available to indicate that time is an important variable. Since the sorption of both fractions of starch increases with time, the farther back in the papermaking process that starch is added, the more time there will be available for retention by sorption. The most logical point of application would be in the beaters. Other investigators (*2, 28–30*) have shown, however, that starch added at the beaters loses much of its effectiveness in improving strength properties if subjected to much refining action. Therefore, a compromise should be made between time for sorption and possible deleterious effects of refining operations. The rate of sorption can be increased by increasing the amount of starch added.

Retention by sorption is greatly enhanced at low temperatures. Mill operators can take advantage of this fact in the wintertime when the water supply is cold and usually has to be warmed for papermaking purposes. If the starch can be added to the pulp in cold water before extreme dilution, a large amount of starch, especially the amylose fraction, will be sorbed. Later warming of the pulp-starch-water system will remove only a small amount of the sorbed starch because of the large temperature hysteresis effect.

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ASSOCIATION NEWS AND EVENTS

Inter-Society Color Council

The annual meeting of the Inter-Society Color Council will be held at the Statler Hotel, New York, N. Y., on Feb. 7–9, 1952. The Technical Association of the Pulp and Paper Industry is a member of the Color Council. Its Members are welcome to attend this meeting.

The papers to be presented include the following:

Color of Oils, by Proctor Thomson
Functional Approach to Color, by Faber Birrer
Color Reproduction, by Arthur C. Hardy
Color in Color Television, by Peter C. Goldmark
Seeing Color Differences, by W. D. Wright
Color in Color Photography, by Ralph M. Evans
Color Appearance, by Harry Helson
Color in Merchandising, by Kenneth C. Welch
Abstract Movies, by A. H. King
Right Color—Wrong Answer, by Gladys Muller
Color in Textiles, by E. I. Stearns
Artists and Their Colors, by Martin Fischer
Color Phenomena, by I. A. Balinken

A number of the member societies and the ISCC will have interesting exhibits.

Mechanical Pulping Committee Meeting

F. W. O'Neil of the College of Forestry, State University of New York, Syracuse, N. Y., and Chairman of the TAPPI Mechanical Pulping Committee announces that at the meeting of his committee Monday afternoon, February 18, at the Commodore Hotel, during the TAPPI Annual Meeting, consideration will be given to the formation of a Subcommittee on Semichemical Pulping. Members of the Association interested in groundwood and semichemical pulping are welcome to attend.

Papermaking (Fourdrinier) Committee

Noel R. Phillips of the Champion Paper & Fibre Co. and Chairman of the TAPPI Papermaking (Fourdrinier) Committee advises that two "Reservation and Working Seminar" meetings will be held during the TAPPI Annual Meeting. One will deal with Paper Machine Shake and the other with Paper Defects.

Attendance at these meetings will be limited to individuals employed by mills operating fourdrinier machines. Individuals who desire to attend should write to R. G. Macdonald, TAPPI, 122 E. 42nd Street, New York 17, N. Y., and request a copy of the questionnaire which will be used as a basis for the round-table discussions. The questionnaire should be filled out and returned to Mr. Macdonald prior to the meeting. Companies that have already returned questionnaires on paper machine shake are eligible to send representatives to the February meeting.

Papermaking Cylinder Committee

During the 1952 Annual Convention of TAPPI (Feb. 18–21, 1952) the Cylinder Committee is planning to hold two "Reservation and Working Seminars." James J. Harrison of the Michigan Carton Co., Battle Creek, Mich., is Chairman of the Committee.

Attendance at these meetings will necessarily have to be limited due to the nature of the subjects to be covered, as well as the desire of the Committee to have these meetings conducted on a strictly informal discussion basis.

Requirements for attendance at these meetings have been set up as follows:

1. The individual must be employed by a mill that is operating Cylinder Machines.
2. The individual should write R. G. Macdonald, Secretary-Treasurer of TAPPI, 122 E. 42nd Street, New York 17, N. Y., signifying his intentions of attending one or both meetings and of participating in the discussions before Feb. 1, 1952.
3. Upon receipt of the individual's letter he will be forwarded a questionnaire that will serve as the basis of the discussion at the meeting. It is suggested that the individuals fill out the questionnaires and be prepared to discuss their various phases at the meeting.
4. Individuals who have complied with the first three conditions will be eligible to attend the meetings.

The Reservations meetings to be held during the 1952 Convention are listed below:

1. Cleaning and Refining of Wastepapers for use on Cylinder Machines.
2. Wet End Forming and Pressing on Cylinder Machines.

From the information developed through the questionnaire and the meetings, the Cylinder Committee hopes to be able to arrive at some of the fundamentals of Cylinder Machine Operation.

The identity of the individuals and the mills who respond and take part in the meetings and answering questionnaires will remain confidential.

PERSONAL MENTION

Items About New and Old Tappimen

New TAPPI Members

Luiz Andrade, Mill Owner and Manager, Industria de Papelao e Caixas Andrade S/A, Est. Sao Paulo, Brazil. Attended Sao Paulo College.

Carl Hj. Backstrom, Works Superintendent, Stromsbruks A/B, Stromsbruk, Sweden, a 1932 graduate of Abo Akademi, Finland.

Tryggve E. E. Bergek, Research Manager, Billeruds A/B, Saffle, Sweden, a 1937 graduate of the Royal University of Technology, Stockholm.

R. William Brown, Jr., Plant Engineer, St. Regis Paper Co., Howland, Me., a 1942 graduate of Clarkson College of Technology.

John R. Callaham, Editor, *Chemical Engineering*, New York, N. Y., a 1933 graduate of the University of South Carolina.

Charles E. Cannon, Plant Superintendent, National Container Corp., Container Div., Tomahawk, Wis.

Robert E. Cates, Technical Service Engineer, Hercules Powder Co., Wilmington, Del., a 1948 graduate of the University of Tennessee.

Anthony J. Cirrito, Development Engineer, Lukens Steel Co., Coatesville, Pa., a 1939 graduate of Villanova College.

Mario J. de la Vega, Mill Superintendent, Compania Papelera "El Fenix" S. A., Mexico City, Mexico, a 1941 graduate of the National University.

Phillip W. Estes, Assistant Chemist, International Paper Co., Chisholm, Me., a 1949 graduate of Bowdoin College.

Harald Fiebiger, Assistant Manager and Chief of Laboratory, Papier und Pergamentpapierfabrik Seltmans GmbH., Bavaria, Germany, a 1950 graduate of the Technical University of Breslau.

Janine H. Fourcaut, Technical Manager, Societe Professionnelle des Papiers de Presse, Paris, France. Attended the University of Paris.

John Fuko, Chief Chemist, River Raisin Paper Co., Monroe, Mich., a 1944 graduate of Wayne University.

Jean Gerard, Vice-President, Societe de Chimie Industrielle, Paris, France. Attended the Institut de Chimie, Nancy.

Libero Gori, Director, Paper Dept., Instituto Peligrafico delle State, Rome, Italy, a 1923 graduate of Pavia College.

George P. Guyon, Manager, Cartonneries de la Rochette, Nancy, France.

Victor P. Head, Director, Hydraulic Research, Fischer & Porter Co., Hatboro, Pa., a 1941 graduate of Rensselaer Polytechnic Institute.

Gilbert L. Heath, Production Manager, Scott Paper Co., Chester, Pa., a 1937 graduate of Pennsylvania State College.

Robert T. Holt, Acting Chief Chemist, National Container Corp. of Virginia, Big Island, Va., a 1950 graduate of Newberry College.

Edgar J. Justus, Control Engineer, Beloit Iron Works, Beloit, Wis., a 1947 graduate of Georgia School of Technology.

Pyara L. Kapur, Master General of Ordnance, M.G.O. Publications Section, Army Headquarters, New Delhi, India.

Sten G. Lindberg, Resident Manager, Billeruds A/B, Saffle, Sweden, a 1928 graduate of Chalmers Technical University.

Albert E. Logan, Section Head, Buckeye Cotton Oil Co., Cincinnati, Ohio, a 1933 graduate of the University of Colorado with an M.S. degree in 1934.

John M. MacBrayne, Chief Industrial Engineer, Union Bag & Paper Corp., Savannah, Ga., a 1931 graduate of Massachusetts Institute of Technology.

Lawrence D. Mailloux, Vice-President and Sales Manager, Original Bradford Soap Works, Inc., W. Warwick, R. I., a 1928 graduate of Providence College with a Ph.B. degree.

Robert A. McDowell, District Manager, Reliance Electric & Engineering Co., Cleveland, Ohio, a 1921 graduate of Case College.

Oquinena Morales, Paper Mill Technician; San Sebastian, Spain. Attended the Polytechnic Institute, Grenoble, France.

Joseph P. Nugent, Manager, U. S. Machinery Co., Inc., New York, N. Y., a 1932 graduate of the University of Notre Dame.

Carl-Filip Palm, General Manager, Zapparfors Co., Norrsundet, Sweden, a 1916 graduate of the Technical Institute.

Edwon M. Peters, Sales Engineer, The Bristol Co., Seattle, Wash., a 1938 graduate of the University of Washington.

Ronald C. Povey, Factory Manager, John Dickinson & Co. Ltd., Home Park Mills, King's Langley, Herts, England.

Lowell O. Sell, Technical Assistant, The Institute of Paper Chemistry, Appleton, Wis., a 1950 graduate of Lawrence College.

Raymond W. Snyder, Student, New York State College of Forestry, Syracuse, N. Y.

Charles P. Spalding, Sales Engineer, Beloit Iron Works, Beloit, Wis., a 1917 graduate of Bowdoin College.

Marion C. Steinmann, Librarian, Rochester Institute of Technology, Rochester, N. Y., a 1925 graduate of the University of Rochester.

Siegfried Studer, Superintendent, Papierfabrik Utzenstorf, Utzenstorf/BE, Switzerland, a 1942 graduate of Zurich College with a D.Sc.Nat. degree.

Sun, Cheng Mo, Assistant General Manager, Tatu Paper Mill, Tatu, Taiwan, China, a 1924 graduate of Ohio State University with an M.S. degree in 1925.

A. Takahashi, Chief Chemist, Research Laboratory, Hokuetsu Paper Mills Ltd., Nagaoka City, Japan, a 1927 graduate of Nagaoka Technical College.

Maxwell Tuck, Research Chemist, Howard Smith Paper Mills Ltd., Cornwall, Ont., Canada, a 1945 graduate of the University of Alberta, with a Ph.D. degree from McGill University in 1947.

Edward H. Ungewitter, Assistant Plant Engineer, St. Marys Kraft Corp., St. Marys, Ga., a 1938 graduate of the University of Connecticut.

Thomas W. Waldrop, Instrumentation Engineer, Republic Flow Meters Co., Atlanta, Ga., a 1943 graduate of Alabama Polytechnic Institute.

Marshall O. Walker, Chief Chemist, Michigan Abrasive Co., Detroit, Mich., a 1938 graduate of the University of Buffalo.

Spencer H. Watkins, Research Chemist, Hercules Powder Co., Wilmington, Del., a 1950 graduate of the University of Wisconsin, with a Ph.D. degree.

James Winn, Plant Engineer, Anglo-Canadian Pulp &

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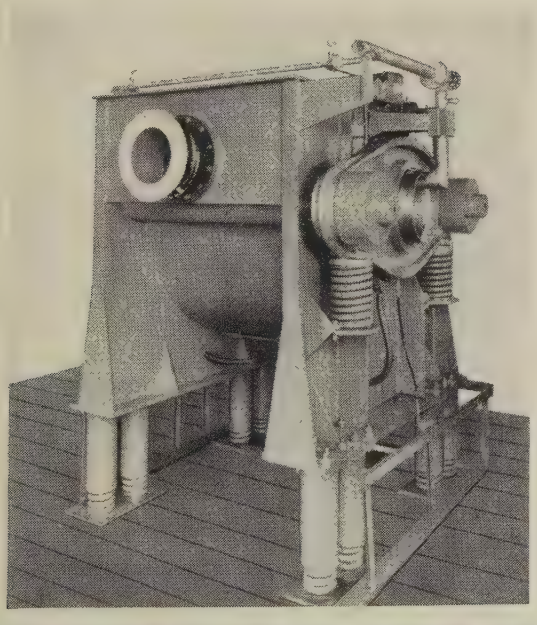

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Paper Mills Ltd., Quebec City, P. Q., Canada, a 1935 graduate of McGill University.

Kenneth M. Winrich, Chief Chemist, Cornell Paperboard Products Co., Cornell, Wis., a 1934 graduate of the University of Wisconsin.

TAPPI Notes

John L. Aldrich, formerly of Cellulose Fibres, Inc., Holyoke, Mass., is now Captain, U. S. Air Force, Westover Air Force Base, Mass.

Richard G. Anderson, formerly of Thiele Kaolin Co., Sandersville, Ga., is now Chemical Engineer for W. H. & L. D. Betz, Philadelphia, Pa.

E. E. Archibald, Jr., formerly of Hollingsworth & Whitney Co., Mobile, Ala., is now Supervisor of Process and Quality Control, Pulp & Paper Division, Potlatch Forests, Inc., Lewiston, Idaho.

Berle B. Babler, formerly of Fleming & Sons, Dallas, Tex., is now Technical Director of the Atlanta Paper Co., Atlanta, Ga.

Ernest F. Barker, formerly of the Sorg Paper Co., Middletown, Ohio, is now Senior Chemist for the Oxford Miami Paper Co., W. Carrollton, Ohio.

Kshiroda K. Baruwa, formerly Student of the University of Maine, Orono, Me., is now Assistant Chemist for the St. Regis Paper Co., Howland, Me.

LeRoy J. Bauer, formerly of the Black-Clawson Co., Hamilton, Ohio, is now Manager of the Pulp and Paper Division, the Pierce Consulting Engineering Co., Boston, Mass.

G. Burkhard, formerly of the St. John Sulphite Co., St. John, N. B., is now Head of Research for the Quebec North Shore Paper Co., Baie Commeau, P. Q.

Claud Christiansen, formerly of Potlatch Forests, Inc., Lewiston, Idaho, is now with the Pulp and Paper Research Institute of Canada, Montreal, P. Q.

Russell L. Clark, formerly of the Newton Falls Paper Co., Newton Falls, N. Y., is now Chemist for the Mead Corp., Chillicothe, Ohio.

R. F. Cuyle of the Hollingsworth & Whitney Co. has transferred from Mobile, Ala., to Boston, Mass., where he is Assistant to the Vice-President.

Peter F. Della Luna is now Superintendent of the Stevens Paper Mills, Inc., Westfield, Mass.

Arthur G. Dreis, formerly of the PMC Dept., Hercules Powder Co., is now Director of the Products Development Dept., P. H. Glatfelter Co., Spring Grove, Pa.

Leslie A. Dow is now in the Technical Service Dept., Eastern Corp., Lincoln, Me.

Pierre Drewsen, formerly member of the TAPPI Executive Committee, was elected Mayor of Northampton, Mass., in the recent G.O.P. sweep in that city. Dr. Drewsen was at one time Technical Director of Hinde & Dauch Paper Co., Sandusky, Ohio.

George A. Farrah is now Assistant to the Vice-President in Charge of the Wisconsin-Minnesota area of the National Container Corp., Tomahawk, Wis. He was formerly located at Long Island City, N. Y.

Clifford A. Flanders, formerly Assistant Biochemist at West Virgina University, Morgantown, W. Va., is now Director of the Chemical Products Division, Fisher Scientific Co., New York, N. Y.

P. A. Forni, formerly Sales Engineer for P. J. Schweitzer, Inc., Lee, Mass., is now Deputy Chief, Pulp and Paper Branch, Economic Cooperation Administration, Washington, D. C.

Gosta Genberg, formerly of the Swedish Cellulose Co., Stockholm, is now Chemical Engineer with Kastrup, Gurberg & Co. A/B, Stockholm, Sweden.

Nelson F. Getchell, formerly of the Ludlow Mfg. & Sales Co., Boston, Mass., is now with the National Cotton Council of America, Washington, D. C.

Raymond S. Hatch, formerly Consultant of Monterrey, Calif., is now Acting Director of Research, Hudson Pulp & Paper Co., Palatka, Fla.

Emil Heuser, Consultant of La Jolla, Calif., recently gave a two-day series of lectures to the chemical research staff of Olin Industries, Inc., E. Alton, Ill. He discussed the growth of cellulose knowledge and the present status of cellulose chemistry.

H. Wyatt Johnston, formerly Vice-President of Sutherland Refiner Ltd., has established a private consulting engineering practice at 4048 Gage Road, Montreal 6, P. Q. Sutherland Refiner Ltd. has retained him as a consultant.

Frank D. Libby is now Pulp and Paper Consultant for the Kalamazoo Vegetable Parchment Co., Kalamazoo, Mich. He was formerly Manager of Manufacture.

David E. MacKnight is now Technical Director for the Alpha Cellulose Corp., Bloomington, Ill.

R. J. MacMeekin, Chief Engineer of Economy Pumps, Inc., has transferred his office from Hamilton, Ohio, to Philadelphia, Pa.

James McClure is now Director of Plant Operations, Sherman Paper Products Co., Chicago, Ill.

W. R. McKelvy, formerly of the Patchogue-Plymouth Mills, Patchogue, N. Y., is now Manager of Hazelhurst Mills, Hazelhurst, Ga.

Robert Magruder, formerly Groundwood Superintendent, St. Regis Paper Co., Deferiet, N. Y., is now with the Paper Corp. of America, Cheboygan, Mich.

J. C. Nutter is now Assistant Mills Manager, Gair Co., Canada, Ltd., Toronto, Ont.

David F. Pollard, formerly of the Great Northern Paper Co., Millinocket, Me., is now Engineer for S. D. Warren Co., Cumberland Mills, Me.

Richard W. Porter, formerly Editorial Director of the *Paper Industry*, Chicago, is now Editor of *Fibre Containers*, Chicago, Ill.

Harry A. Raddin, formerly Chemical Engineer for Miller & Collins, Inc., is now with Hofft Co., Inc., Richmond, Va.

James F. Sheehy, Vice-President of Rayonier, Inc., has transferred his office from Hoquiam, Wash., to New York City.

Gorham G. Simmons, formerly of the Berwin Paper Mfg. Co., Dansville, N. Y., is now in charge of stock preparation control, Central Paper Co., Muskegon, Mich.

S. G. Slifkin, formerly of Reproduction Products, Franklin Park, Ill., is now a Technical Consultant, 35 N. Northwest Highway, Palatine, Ill.

Wesley N. Stickel is now Technical Supervisor of Texon, Inc., S. Hadley Falls, Mass.

L. V. Subba Rao is now Paper Technologist for K.I.C. Ltd., Vuyur, Kistna District, India.

Roy P. Tidman, formerly of the West Virginia Pulp & Paper Co., Covington, Va., is now Engineer for the Scott Paper Co., Chester, Pa.

Pekka Vakomies, formerly Superintendent of Sunila Co., Sunila, Finland, is now Design Engineer for the Irving Pulp & Paper Co., Fairville, N. B.

Vernon E. Varhus is now Chief Engineer for General Mills, Inc., Keokuk, Iowa. He was formerly Development Engineer at Minneapolis.

F. B. Wardwell, formerly Research Chemist for the Brown Co., Berlin, N. H., is now a Captain in the U. S. Marine Corps, Flying Fighters, Squantum, Mass.

Josiah Wood, formerly of E. Long Ltd., is now Sales Engineer for Sherbrooke Machineries Ltd., Montreal, P. Q.

Milton Zucker, Research Manager for Interchemical Corp., has been transferred from Elizabeth, N. J., to New York City.

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J. N. Stephenson, Editor-in-Chief of the *Pulp and Paper Magazine of Canada*, Gardenvale, P. Q., recently reviewed the list of members of TAPPI who joined in 1915 (Charter Year), of which he was one. The others checked by him are: W. E. B. Baker, F. C. Clark, F. A. Curtis, M. J. Davis, J. F. Davis, H. D. Eliason, D. C. Everest, F. W. Farrell, V. E. Fishburn, H. M. Fletcher, R. C. Griffin, A. B. Green, H. W. Guettler, R. S. Hatch, A. J. Haug, M. W. Hedden, E. M. Mahler, R. M. McKee, G. H. Mead, T. R. H. Murphy, P. A. Paulson, C. Patch, R. B. Robertson, J. D. Rue, H. J. Skinner, A. D. Spierling, J. N. Stephenson, E. Sutermeister, and S. D. Wells.

* * *

A. B. Moody has succeeded W. J. Pilz, retired, as the official corporate representative of the Everett Pulp & Paper Co., Everett, Wash., in TAPPI.

Sergio H. Magalhaes Anturies has succeeded Ermani Coutinho da Costa as the official corporate representative in TAPPI of Cia. Ind. de Papel Pirahy, Rio de Janeiro, Brazil.

The name of the H. R. MacMillan Export Co., Ltd., Pulp Division, has been changed to MacMillan & Bloedel Ltd., Pulp Division, Nanaimo, B. C. C. Crispin is the official corporate representative in TAPPI.

* * *

Companies that have recently become sustaining members of the Technical Association are the following:

Albany Felt Co., Albany, N. Y.
American Key Products, Inc., New York, N. Y.
Clark-Cukin Co., Lee, Mass.
Earl Paint Co., Utica, N. Y.
Ebasco Services, Inc., New York, N. Y.
H. K. Ferguson Co., Cleveland, Ohio
Howard Publishing Co., Chicago, Ill.
John Inglis Ltd., Toronto, Ont.
Magnus Chemical Co., Garwood, N. J.
Ranney Method Water Supplies, Inc., Columbus, Ohio
Rotoreaed Corp., Bronxville, N. Y.

Industry Notes

Preliminary negotiations for pulp from Europe in 1952 indicate that foreign sources will allot about the same tonnage as during the last half of 1951. If the OPS decides to roll back the present ceiling prices on imported pulp the prospect of more pulp from Europe is dim.

* * *

The Manistique Paper Mill of Manistique, Mich., has been purchased from the Mead Corp. by the Trenton *Times*, Trenton, N. J. Production of 25,000 tons annually will be converted to newsprint, all of which, it is reported, will go to South American publishers. The selling price was about four million dollars.

* * *

Amendment 1 to M-69 limits users of sulphur to inventories of 25 days' supply or a practical working minimum, whichever is less. However, users who customarily get single shipments of sulphur amounting to three or four months' supply at normal production rates will be allowed to get the large shipments provided that after receipt of such shipment no additional delivery is permissible as long as the inventory continues to exceed requirements for the next succeeding 25 calendar days.

* * *

The Defense Production Administration has announced that it would grant certificates of necessity to facilitate a proposed expansion of the domestic newsprint industry of 494,000 tons. The DPA does not propose, however, to take immediate action on manufacturers' applications because of other types of expansion projects vital to defense, and the critical shortage of structural steel. It is expected that the new projects will be in production between mid-1953 and some time in 1955.

* * *

OPS has issued Amendment 4 to SR 12 of CPR-22. The effect of this amendment is that paper manufacturers who have been pricing under GCPR may continue to do so indefinitely. Those manufacturers who have been pricing under CPR-22, or who have tailored orders, will not be affected. Manufacturers who have been pricing under GCPR, but who may wish to go under CPR-22 or SR 17, may do so. However, if a manufacturer elects to price under CPR-22 or SR 17, he must do so for all his products and the election to do so is final.

* * *

James L. Madden, President of Hollingsworth & Whitney Co., has been elected President of the American Forest Products Industries, Inc., succeeding N. F. McGowan of the W.T. Smith Lumber Co.

* * *

William H. Chisholm, who has served as Deputy Director in the Pulp, Paper and Paperboard Division, NPA, during the past several months, has returned to his position as Vice-President of the Oxford Paper Co. Mr. Chisholm joined the Division in May and first served as Special Assistant for Operations in the Division.

* * *

Llewellyn (Lew) Nicolson, President of the D. C. Paper Mills, Washington, D. C., has been promoted to the position of Deputy Director of the Division. Mr. Nicolson joined the staff of the Division in May and has served as Chief of the Wood Pulp Section until the present time.

* * *

J. Prentis Sanger, Assistant Sales Manager, Crocker, Burbank & Co. at Fitchburg, Mass., has been appointed to serve as Special Assistant for Operations. Mr. Sanger is on loan from his company.

* * *

John J. Steinbinder, Assistant Machine Manager of Riegel Paper Corp., Milford, N. J., is being loaned by his company to serve as Chief of the Chemical Section of the Division. He replaces Harold H. Heller, who was on loan from Kimberly-Clark Corp., Neenah, Wis.

* * *

William McNair, Pulp Division of St. Regis Paper Co., is being loaned to serve as Chief of the Wood Pulp Section of the Division (Source: NPA).

* * *

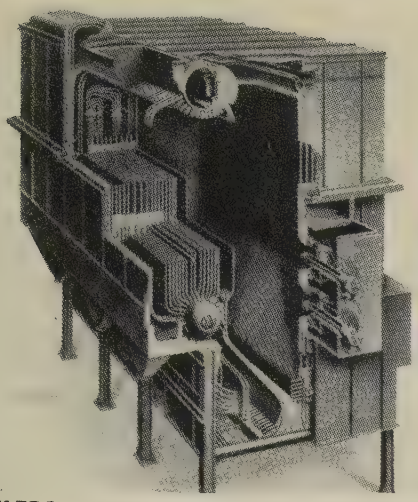
The new ceiling prices for pulpwood in Pennsylvania and southern New York are as follows:

Pine, peeled	$18.50 per cord
Pine, rough	14.00 per cord
Hardwood, peeled	17.50 per cord
Hardwood, rough	13.00 per cord
Hemlock, peeled	17.00 per cord
Hemlock, rough	12.50 per cord

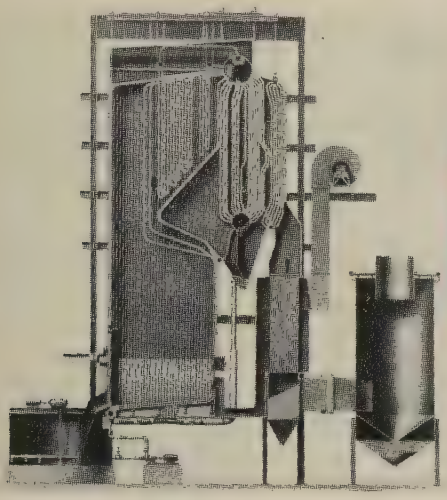
The established differential between the prices for peeled and rough pulpwood is $4.50.

* * *

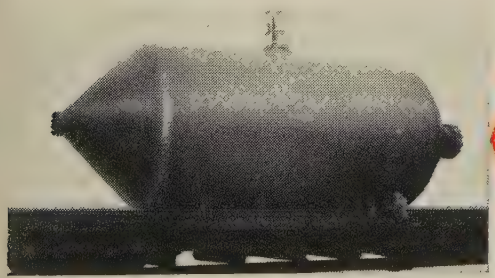
The production of shipping sack kraft paper in 1951 exceeded 1950 production by about 35,000 tons. Production in 1951 averaged 197,400 tons per quarter. It is expected



BOILERS—B&W boilers have a long and outstanding record for low-cost steam generation in pulp and paper mills. They span a range of types and sizes to satisfy every requirement of capacity, space, temperature, pressure, fuel, and method of firing. Each type combines the dependability of job-proved design with every economy of standardization commensurate with flexibility.

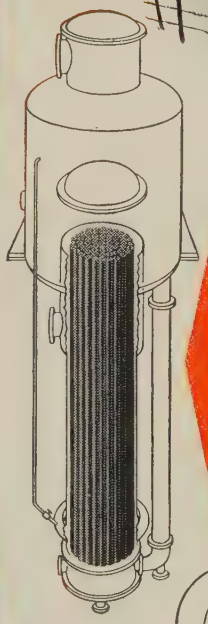


RECOVERY UNITS—Efficient chemical recovery and reduction, along with maximum steam generation per ton of pulp, are combined with economical operation and maintenance in B&W Recovery Units. Installations for burning waste liquors of the kraft, soda, and bisulphate (magnesium, calcium, and ammonium) processes have a total recovery capacity of over 16,000 tons. B&W recovery units were the first provided with automatic soot blowers to eliminate routine hand lancing.



PRESSURE VESSELS—Dependable welded processing units, in sizes and shapes for any paper mill requirements, are fabricated by B&W from carbon, alloy, or clad steels. All vessels are x-ray inspected and stress relieved.

time-proved aids to LOW COST production



TUBES—Seamless and Welded steel tubes are made by B&W in a range of carbon, alloy, and stainless steel analyses to satisfy every requirement for condensers, evaporators, heat exchangers, recovery units, boilers, and other paper mill applications. Modern improvements in pulp and paper processes impose a greater variety of tubing requirements than ever before. B&W Tube Representatives offer a wealth of experience on tubing problems to help users determine the analyses best suited for specific operating conditions.

B&W BABCOCK & WILCOX

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that 1952 production will be sufficient to meet all normal requirements.

* * *

The biggest thing in the book publishing industry is now under way in New York City. The Revised Standard Version of the Holy Bible, being published by Thomas Nelson & Sons, will have a first run of one million copies. While some government pamphlets and handbooks, children's books, or comic books have been ordered in greater numbers, the materials and costs involved in this printing, undoubtedly make this the biggest project in book publishing history.

Linotype operators in the composing room of H. Wolff Book Mfg. Co., New York, have already begun setting the 1344-page book, and when this is finished, presses at the American Book Co., New York; the Riverside Press, Cambridge, Mass.; and H. Wolff will run from steel-faced electros for six or seven months to complete the job. Publication is scheduled for September, 1952.

An indication of the size of the publishing task being undertaken can be seen in the fact that the printing will require more than 1000 tons of paper and 16,670 pounds (2000 gallons) of ink. It will also use 140 tons of binders board, 10 tons of type metal, 71 1/2 miles of 40-inch cloth, and 18,750,000 yards of thread.

* * *

The P. H. Glatfelter Co., Spring Grove, Pa., salvaged more than 328,000 pounds of iron scrap in 1951 for the steel industry. For every pound of steel produced a half pound of scrap is needed.

* * *

The 21st National Packaging Exposition will be held in the Atlantic City Auditorium on April 1–4, 1952. This exposition is sponsored by the American Management Association.

* * *

Construction work is soon to be started on a new pulp pilot plant for the Brown Co., Berlin, N. H. It is expected to be far superior to any other pilot plant in the wood pulp industry. It will be a complete pulp mill on a small scale. It will enable the company to extend its studies of the various advanced techniques of preparing pulps without interfering with present mill operations. Included in the plant will be a stainless steel digester which will cook both sulphite and sulphate pulps, a combination washer and thickener of modern design, a mixer, a bleachery, and a pulp refining system. It will be under the direction of the Research and Development Department.

* * *

Ways and means to purchase and use ledger papers and index cards more efficiently for bookkeeping, accounting, and other record keeping are described in a new booklet issued by Parsons Paper Co., Holyoke, Mass. One of the most interesting features of the booklet is a table indicating how many handlings and how many years of service may be expected of the various grades of cotton fiber paper.

* * *

Figures presented at the annual meeting of the National Paperboard Association indicate that the total production of paperboard for 1951 will be over 11,700,000 tons, a total appreciably higher than any past year.

* * *

The Sulphite Pulp Manufacturers League has established a $10,000 research project at the University of Wyoming to investigate spent sulphite liquor in search for a big volume use for it as a binder. Fields in which this utilization seem a good possibility, aside from fuel briquetting are processing of iron ore, foundry cores, and a variety of surfacing applications for roads more highly developed than the gravel surfaces on which it is already widely used in Wisconsin.

At the pilot plant in Appleton a test run of several months' duration is in operation, using a new evaporator of a design never before used for concentrating spent liquor.

Present plans are to keep the trial going continuously for at least three months. If the new evaporator keeps on operating successfully and if its principles prove to apply to spent sulphite liquor, this will give the Wisconsin pulp and paper industry a second useful method for concentrating sulphite liquor so that its wood solids can be burned under a boiler. Until now, the only evaporator in this state capable of removing excess water from this material without clogging up with lime scale has been a Swedish-made reverse-flow unit that has been running almost continuously for two years in the League's pilot plant here. All Wisconsin progress in this field has been dependent on the output of this single evaporator.

The new evaporator, made after extensive laboratory tests by Struthers Wells Corp. of Warren, Pa., operates on a simple principle entirely different from the Swedish unit. Instead of washing the lime scale off after it has crystallized on evaporator surfaces, the Struthers Wells equipment provides a bed of lime salts where lime can crystallize without harming anything. Thus far, there has been no indication of fouling.

The new test unit has a capacity rated to evaporate 500 pounds of water per hour, employing a single effect. This is 25% of the pilot plant's Swedish evaporator capacity, also single effect. A mill-size industrial application of the new principle should for economical operation employ a quadruple effect, according to Struthers Wells engineers.

To evaporate to fuel consistency with quadruple effect the total outflow of sulphite liquor from a sulphite pulp mill the size of those along the Fox and Wisconsin Rivers would require four sets of evaporators, each set having about 125 to 150 times the capacity of the unit now being tested, League technologists calculate.

* * *

A carnauba substitute designed especially for use in carbon paper manufacture, has been developed by the Concord Chemical Co., Moorestown, N. J. The main selling point of the new product, trade-named Parawax, is its low cost.

* * *

Monadnock Paper Mills, Inc., Bennington, N. H., has taken an option for purchase of a 75-acre site in Elizabethton, Tenn. H. K. Ferguson Co. have been selected as engineers to design and erect a pulp mill. The citizens of Elizabethton have approved a 20 million dollar industrial bond issue for a plant to be leased to Monadnock.

* * *

A new Manual on Preservation, Packaging and Packing of Military Supplies and Equipment has been issued by the Department of Defense. Copies may be obtained from the Government Printing Office, Superintendent of Documents, Washington 25, D. C. Request should include the title and one of the following reference numbers: Department of the Army Technical Manual TM 38-230, Department of the Navy Manual NavExos p-938, or Department of the Air Force Manual AFM 71-1.

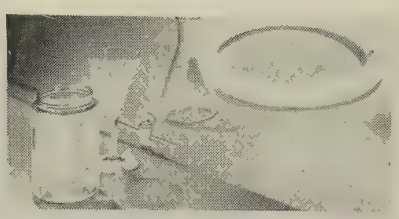
* * *

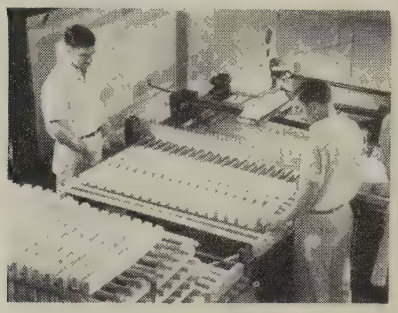
The General Electric Co., Schenectady, N. Y., has issued a 1952 Catalog (GEC-1016) on Measuring Equipment for Laboratory and Production Testing.

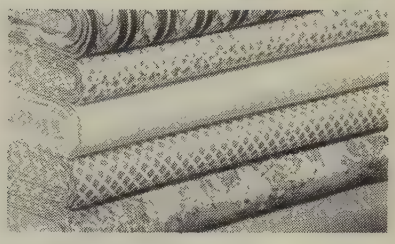
* * *

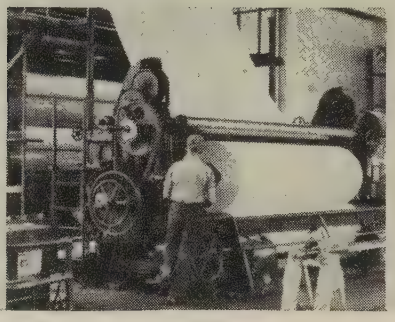
The Gaylord Container Corp., St. Louis, Mo., has acquired the Fairfield Paper & Container Co. with mills in Baltimore,

NEED ALKALIES?









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Monochlorobenzene
Methanol
Formaldehyde
Ammonium Chloride

Ohio, and Beaver Falls, Pa., and the Dresden Paper Mills Co., Dresden, Ohio. Gaylord operations now include three paper mills and seventeen converting plants.

* * *

More polyethylene is now available for civilian use. Du Pont's alterations in manufacturing procedures have increased that firm's output. The new plant of Bakelite is now operating. The output of polyethylene has been running at a rate in excess of 60 million pounds a year. Of this about 65% is available for civilian use. Packaging products now take 30% of the supply. Drum liners for chemicals and pharmaceuticals have been put on the essential list. Film, tubing, and wax mixes for frozen foods are obtaining increasing amounts.

* * *

Olin Industries, East Alton, Ill., and Frost Lumber Industries, Shreveport, La., have agreed on a plan to consolidate their properties and business. The Frost Industries control 450,000 acres of woodlands. It is expected that a mill to manufacture dissolving pulps will be built on the properties.

* * *

Simpson Logging Co., Shelton, Wash., has purchased the assets of the Everett Pulp & Paper Co., Everett, Wash. A. B. Moody will be general manager and W. J. Pilz will retire.

* * *

A second newsprint machine will be installed in the mill of the West Tacoma Newsprint Co., Tacoma, Wash.

* * *

The DPA has announced that the pulp and paper industry in the United States produced more pulp in 1951 than in 1950 despite reduced supplies of sulphur. This was due to improved technical efficiencies and conservation measures.

* * *

Ralph M. Lindgren, who has directed the research of the U. S. Division of Forest Pathology in the Gulf States since 1947, has been transferred to the Forest Products Laboratory, Madison, Wis., where he will undertake supervision of research on decays, stains, and other fungus defects of wood products.

* * *

Edward Hartshorne has been appointed Manager of the Research & Development Dept., Olin Cellophane Division, Olin Industries, Inc., at New Haven, Conn.

* * *

International Nickel Co., 67 Wall St., New York 5, N. Y., has issued a 32-page bulletin on Nickel Alloy Castings in Industry.

* * *

The General Electric Co., Schenectady, N. Y., is offering a 12-minute, black and white sound slide film on the subject of Productive Maintenance for showing by interested groups. Arrangements for showing can be made through any of the company's apparatus sales offices or service shops.

* * *

Foster Wheeler Co., 165 Broadway, New York 6, N. Y., has issued a 20-page booklet on Series AG Package Steam Generators With or Without Superheat.

* * *

The following release of the American Paper and Pulp Association on the effect of certificates of necessity on Federal income from taxes has been sent to pulp and paper companies:

The accelerated amortization program being administered by the Defense Production Administration is based upon the sound accounting principle that the cost of a facility should be absorbed by current operations during the effective life span of that facility. The "effective life span" has always been affected by wear and tear and obsolescence. Wear and tear may not make an installation useless before obsolescence does.

Plants built exclusively for emergency work become obsolete when the emergency passes. Congress, in setting up this program, decided in effect that five years was the correct period for the "effective life span" in which the cost of emergency installations should be absorbed in current operations.

The Defense Production Administration has been issuing "Certificates of Necessity" for such complete installations and such partial installations as, in its judgment, come within the scope of emergency installations. These cover both civilian and military categories.

The effect of this upon the tax and earnings position is not complex. The Government loses no tax income that is legitimately due it, whatever the actuality of the "effective life span" may be in a particular instance.

If the effective life span is five years or less, the Government is in duty and fairness bound to let the facility be absorbed in operating costs.

If the effective life span should be more than five years the results, tax-wise, would be as follows:

By way of illustration let us assume that a proposed new plant will cost $30,000,000 to build and that its normal life-usefulness, according to the Treasury Department, would be 20 years. Assuming no salvage value at the end of the 20 years, that would mean an annual depreciation charge of $1,500,000. If, however, a Certificate of Necessity is granted for 100% of the project, the depreciation charge would be $6,000,000.

These depreciation charges are deducted as part of the cost when computing profits, and current taxes that are assessed on profits will be affected by these depreciation charges.

Let us assume that on the basis of the $1,500,000 depreciation charge the annual profit before taxes is $10,000,000. Then if the Certificate of Necessity were granted, the charge would be $6,000,000 and profit before taxes would be less by $4,500,000, or $5,500,000 instead of the $10,000,000 noted above.

It is obvious that the current taxes on $5,500,000 income would be less than on $10,000,000 income. And that is the basis for much of the hue and cry against the Certificate of Necessity program as "tax evasion."

But is it that? In the first place, would the plant be built without the Certificate of Necessity? It certainly would not if it is known that it will become obsolete in five years.

In the second place, the *income* that escapes taxes in the first five years will surely be assessed for taxes if earned in subsequent years until the plant becomes obsolete.

In other words, the $6,000,000 income that escapes taxes in the first five years will pay taxes for the balance of fifteen years of life-usefulness normally to be expected. This, of course, will be because the entire plant will have been "written off" by the end of the first five years, and there will be no depreciation left to deduct from income thereafter, and in the case cited above, the taxable income would become $11,500,000 instead of $5,500,000.

The only uncertainties are: will there be any earnings after five years? If not, the depreciation charges at the accelerated rates of amortization will have been justified.

Again, if these earnings continue after the five-year period, will the tax return on them be higher, lower, or equal? If equal, there will be no gain either way. But the big question is, will taxes after five years be at higher or lower rates than

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at present? If higher, the company with a Certificate of Necessity will be the loser on net balance because taxes on $11,500,000 instead of $5,500,000 will be assessed at the higher rate. If lower, it will gain a slight advantage. But how many will care to forecast lower income tax rates after the next five years have passed? Not many, I am sure!

The Government cannot lose taxes due to accelerated amortization. Income that escapes taxes in the first five years will be subject to taxes in subsequent years, because of no depreciation charges in those years. The tax on such income over a so-called normal depreciation period will be at least equal, if not more than under the accelerated amortization plan. The Government cannot lose nor can a taxpayer gain under the plan. A taxpayer is simply granted relief, temporarily, and he surely deserves this relief in view of his willingness to take the risk of the investment for defense purposes.

* * *

The office of Price Stabilization has announced dollars-and-cents ceilings for manufacturers on 53 specified grades of writing and other fine papers and also prescribed means of determining ceilings on other grades.

The ceilings, stipulated in Ceiling Price Regulation 91, effective Nov. 10, 1951, apply to an important segment of the paper industry which in 1950 produced 1,200,000 tons of paper valued at $360,000,000, roughly 11% of the domestic production of all kinds of paper exclusive of paperboard.

The spelled-out ceilings are at the same general average level as prices determined under the General Ceiling Price Regulation, GCPR, which froze prices existing in the period Dec. 19, 1950, to Jan. 25, 1951. The dollars-and-cents pricing of major grades will tend to iron out disparities frozen into the price structure under GCPR.

Ceiling prices for paper merchants and distributors will be fixed in a companion regulation which will cover wholesalers' prices to manufacturers, converters, printers, and retailers. Retail ceilings on writing and other fine papers, and, for the time-being wholesale ceilings, are determined under the GCPR or Supplementary Regulation 29 to GCPR.

Papers covered by the new manufacturers' regulation are used in the conduct of social and business correspondence, maintenance of records, and transmission of information, and also include such special papers as blueprint, covering, and cigarette papers. They are made from any one or a combination of pulps produced by chemical or mechanical treatment of rags, flax, and wood fibers, and represent a wide variety of grades and qualities.

Since June 1950, raw materials have advanced sharply in price for the fine paper mills. Cotton, linen, and underwear cuttings are about 70% higher, while domestic bleached sulphite wood pulp is 20% higher and overseas sulphite 90% higher. Sulphite is the principal grade of chemical wood pulp used in the industry.

There have been economies in production offsetting to some extent the higher cost of materials, including higher rates of operation, longer runs, and minimum production of low profit specialties.

Between June, 1950, and the January, 1951, GCPR freeze, manufacturers raised their rag content paper prices from 12 to 33% and their chemical wood pulp paper prices from 12 to 19%.

The spelled-out base prices apply to specific grades listed and their related grades, and, by application of spelled-out differentials, to grades which vary in tint, color, finish, and other particulars. Differentials are also set forth for variations in packaging, trimming, and size of orders.

Following are the base prices spelled out for the key grades of white paper:

Rag Content Bond, ream sealed, trimmed, in standard cartons, per hundredweight: Basis weight 17 pounds or heavier, extra 100% rag $57.50, 100% rag $49.50, 75% rag $38, 50% rag $29.50, 25% rag $24; same, basis weight 16 pounds, extra 100% rag $58.50, 100% rag, $50.50, 75% rag $39, 50% rag $30.50, 25% rag $25.

Rag Content Ledger, ream sealed, trimmed, in cartons, per hundredweight: Extra 100% rag $58.50, 100% rag $50.50, 85% rag $42, 75% rag $39.50, 50% rag $30.50, 25% rag $25.

Rag Content Onionskin and Manifold, ream sealed, trimmed, in cartons or cases: Basis weights 14 to 18 pounds, 17 x 22, per 1000 sheets; 100% rag $10.90, 75% rag $8.80, 25% rag $7.50.

Rag Content Wedding, ream sealed, trimmed, in cartons, basis weight 24 pounds or heavier, per hundredweight: Extra 100% rag $52, 100% rag $49.50, 25% rag $24.

Rag Content Index, sealed, trimmed, in cartons, not heavier than 25½ x 30½—340 M, per hundredweight: 100% rag $50.50, 75% rag $39, 50% rag $30.50, 25% rag $25.

Blueprint, standard wrapped rolls, per hundredweight, carload lots: 100% rag, substance weight 17 pounds $46, substance weight 20½ pounds $45, substance weight 24 pounds $45; 50% rag, substance weight 17 pounds $29.50, substance weight 20½ pounds $28.50; 25% rag, substance weight 17 pounds $24, substance weight 20½ pounds $23. Slightly higher prices are spelled out for less than carload lots.

Rag Negative Paper Base Stock, standard wrapped rolls, per hundredweight, any quantity: Substance weight 14 pounds $61.

Rag Content Cover Papers, machine finish or antique, plain edge, 25% rag, per hundredweight $21.25.

Chemical Wood Pulp Bond, ream sealed, trimmed, in standard cartons, per hundredweight: Basis weight 17 pounds or heavier, air dried (watermarked) $20, No. 1 bond, machine finish (watermarked) $15.50, No. 2 bond, machine finish (watermarked) $14.90, plain bond, machine finish (watermarked) $14; same, basis weight 16 pounds, 60 cents to $1.00 higher.

Chemical Wood Pulp Ledger, ream sealed, trimmed, in cartons, machine finish, per hundredweight: No. 1 ledger (watermarked) $16.50, No. 2 ledger (watermarked) $15.90, plain ledger (watermarked) $15.

Chemical Wood Pulp Onionskin and Manifold, ream sealed, trimmed, in cases or on skids, 17 x 22, per 1000 sheets: Basis weight 7 to 9 pounds, No. 1 watermarked $4.10, No. 2 watermarked $4.00, No. 2 unwatermarked $3.80, plain unwatermarked $3.60; basis weight 10 pounds, 20 cents additional per 1000.

Chemical Wood Pulp Papeteries, Weddings, ream sealed, trimmed, in standard cartons, machine vellum, per hundredweight: Basis weights 24 pounds or heavier, No. 1 $20, No. 2 $17, No. 3 $16.10; same, basis weight 20 pounds, $1.00 additional.

Chemical Wood Pulp Index Bristol, sealed, trimmed, in standard cartons, machine finish, per hundredweight: No. 1 $17, No. 2 $14.70, No. 3 $14.25.

Chemical Wood Pulp Cover Papers, antique or machine finish, per hundredweight: No. 1 plain edge $16.25, No. 2 plain edge $15.25, No. 3 25% plain edge $14.25.

Chemical Wood Pulp Mimeo, sealed, trimmed, in standard cartons, machine finish, per hundredweight: Basis weight over 16 pounds, No. 1 watermarked $15.50, plain unwatermarked $14; same, basis weight 16 pounds, No. 1 $16.20, plain $14.60.

Chemical Wood Pulp Opaque Circular Paper, ream sealed, trimmed, in standard cartons, per hundredweight: Basis weight 17 pounds or heavier $16.15, basis weight 16 pounds $16.85.

Ceiling prices on unclassified grades are the highest prices for sales during either of the following periods: Jan. 1 to June 24, 1950, or Jan. 25 to Sept. 15, 1951.

Classified grades include but are not limited to the following:

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Ceilings on papers which cannot be priced as related or unclassified grades by the foregoing methods may be determined "in line" with prices for the most comparable grade of paper established under the regulation, with the margin over cost no greater than the margin on the comparable grade.

* * *

Wastepaper packers and dealers are quite anxious that present sources of wastepaper should not be "dried up," but that seems to be exactly what is happening in a number of Eastern localities. A great many peddlers, however, are still collecting, but are giving their major attention to scrap metals, which are in extraordinary demand at present, and giving little attention to wastepaper.

The wastepaper industry generally is giving no encouragement to prospective collectors of wastepaper, such as schools, churches, Boy Scouts, etc., for the reason that the dealers are not in a position to dispose of such collections when they are made. Dealers regular sources of supply, they say, are producing more wastepaper than can be sold, and as much as dealers would like to give their support to local enterprises for the raising of funds for charities and civic affairs, it is simply impossible for them to do so at the present time. While everyone is looking forward to the time when wastepaper will again be in demand, just when that will be is difficult to determine now. In the meantime, the wastepaper dealers who are putting out superior-packed bales are enjoying a moderate business.

A growing problem of eliminating certain objectionable kinds of wastepaper faced both packers and mills. Synthetic rubber adhesives, as well as ink bases, containing derivatives or compounds apparently of a similar nature, seem to be driving some of the mill men nearly frantic as the stuff gets into their beaters. Then there is wet-strength kraft appearing in considerable quantities which is baffling to mill men and packers alike. Notice has been served by several paperboard mills that they will reject multiwall bags that have contained an insecticide known as benzene hexachloride on account of its disagreeable odor.

Much attention is being given to the problem by both the consuming mills and the Wastepaper Institute. While the wet-strength kraft and multiwall bags are disturbing to the board mill men, the paper mills are agog over the adhesives used in certain books and magazines, and the ink used in others. Substantial losses have been incurred by dealers and mills in the purchase of papers containing these objectionable substances, and efforts are being made, if not to minimize their use, to make them identifiable to the wastepaper sorters.

Compared with previous full years, including the greatest wartime year, 1944, for both receipts and consumption, here's the way it looks:

Year	*Receipts*	*Consumption*
1944	6,937,000	6,859,000
1946	7,487,000	7,287,100
1947	7,875,000	8,009,000
1948	7,670,000	7,649,000
1949	6,522,400	6,599,600
1950	7,895,000	7,903,600
1951[a]	8,039,000	7,900,000

[a] U. S. Census totals for first 35 weeks; our estimate for last 10 weeks comprises totals for first 45 weeks of 1951.

Mill purchases and consumption during the last three weeks of November and the four weeks of December are expected to bring this year's totals in both columns to close to 9,000,000 tons. They may be slightly under that figure, or may be slightly over it.

* * *

Ten thousand acres of forests have been treated in a regeneration program in Virginia and North Carolina by The Chesapeake Corp. of Virginia. Over 100,000 more eventually will be included in the program, which was started in 1947.

Elis Olsson, chairman of the company, which has one of the largest sulphate pulp mills in the Southeast, began marking and saving seed trees on land being logged in 1918.

Under Chesapeake's current program, merchantable hardwood and pine trees are harvested, with the pine going for pulpwood and the hardwood for cross ties, low grade lumber for pallets, flooring, and hickory handles. Only pine trees are left standing to reproduce. After logging is completed, land clearing crews go through the heavy brush with giant crawler tractors and bulldozers, uprooting and removing second-growth gum and scrub oak trees.

Where no seed trees are available, trees are planted by hand or with machines. Chesapeake officials say that treated areas where seed trees were present have come back in beautiful stands of young pine. They feel confident that their sustained yield program will mean adequate future supplies of pulpwood.

J. H. Johnson is forester for the Chesapeake Corp.

* * *

Construction is well under way on the expansion to the present Hudson Pulp & Paper Corp., Southern Division, Palatka, Fla. mill. This program calls for increasing the capacity of the mill output to a total of 400 daily tons of finished unbleached kraft bag and wrapping papers. Both the pulp and paper mill facilities are being doubled. The pulp mill and the paper mill extension has been laid out so as to provide a separate unit for the preparation of lightweight papers; the existing system to be continued for the manufacture of heavyweight grades.

A water-treating plant, which was not included in the original installation, is being added. It will have a capacity of twelve million gallons per day, served by additional large capacity process water pumps.

The wood yard and wood room facilities are being doubled in capacity with the installation of a new suspended barking drum, log and chip conveyors, chipper, two chip screens, and auxiliary sawdust and refuse conveyors. Three new glazed tile silos will provide ample storage for chips.

In the digester room, there will be five new ten-ton digesters, each arranged with heaters, strainers, and pumps for indirect cooking. There will be a separate gassing-off system for the recovery of turpentine. The present blow-down heat recovery unit will be supplemented to take care of the additional digesters.

A new blow tank equipped with an agitator and tramp iron trap will provide ample storage ahead of the three new 8 by 12-foot countercurrent brown stock washers and deknotters. Auxiliary filtrate and black liquor storage, soap skimming, and foam tanks are located adjacent to the new washroom.

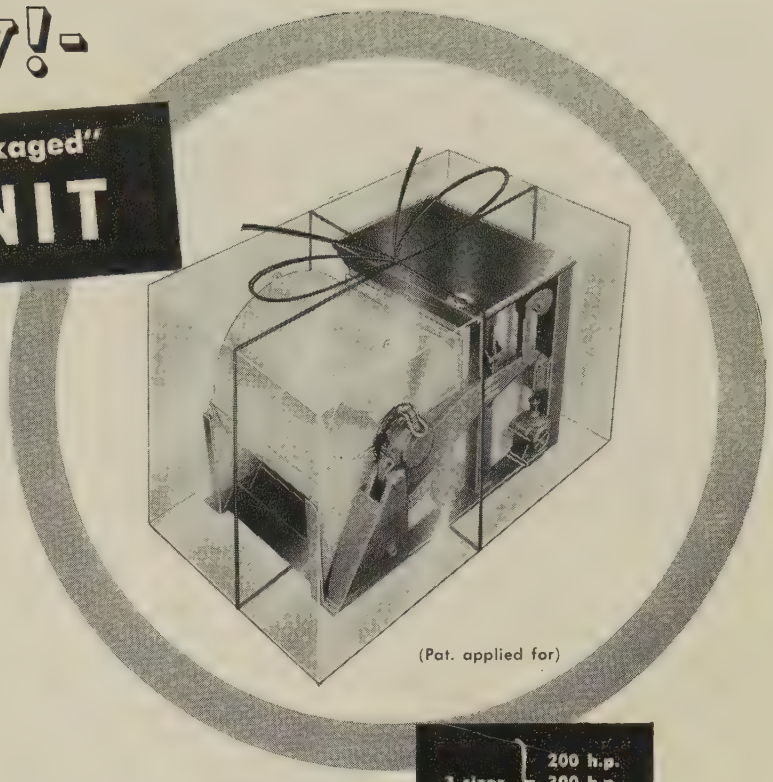
Following the washers are rotary screens, two used as primary screens and one as a secondary screen. Tailings from these flow by gravity to a tailings flat screen, the accepted stock from which is put back into the heavyweight paper system. Screened stock is thickened on a new 8 by 14-foot vacuum decker. The new washed stock and screened stock chests will be of glazed tile construction equipped with propeller type agitators.

The stock preparation room has been laid out for both

Now!-

The first "packaged"

BEATING UNIT



Yes! — a completely pre-assembled Beating Unit . . . that produces better quality, more uniformly refined fibre, at up to 65% savings in power over conventional beaters.

Features: lower installation cost (no assembly in the mill), simpler floor construction, less floor space, lower maintenance — PLUS more refining capacity, greater flexibility of operation, more positive treatment of stock, and no possibility of untreated stock passing through the Beating Unit.

Available for Multibeater (continuous operation) or tub installation. Ask your Jones representative for details.

3 sizes — 200 h.p. 300 h.p. 400 h.p.

Jones

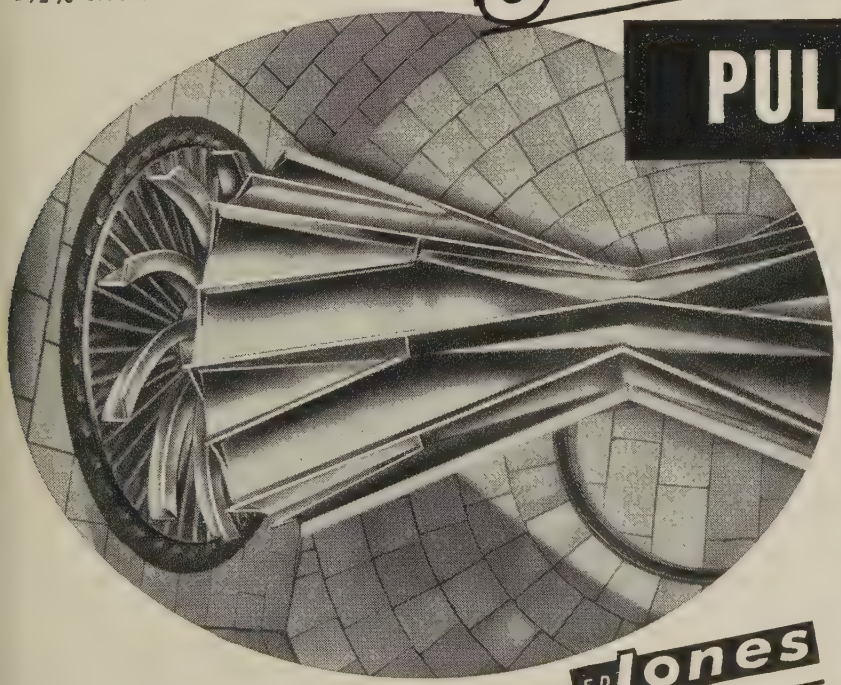
E. D. JONES and SONS COMPANY • PITTSFIELD, MASSACHUSETTS

BUILDERS OF QUALITY STOCK PREPARATION MACHINERY

Rotor of No. 4 size Jones Pulp-Master, shown in tile tank installation in a leading midwestern mill. Capacity 4000 lbs. 6½% stock.

E.D. Jones

PULPING POWER



The rugged, one-piece fabricated rotor of the PULP-MASTER, placed well above the bottom of the tank, gives stock vigorous, continuous circulation — yet allows junk metal to drop to the tank bottom.

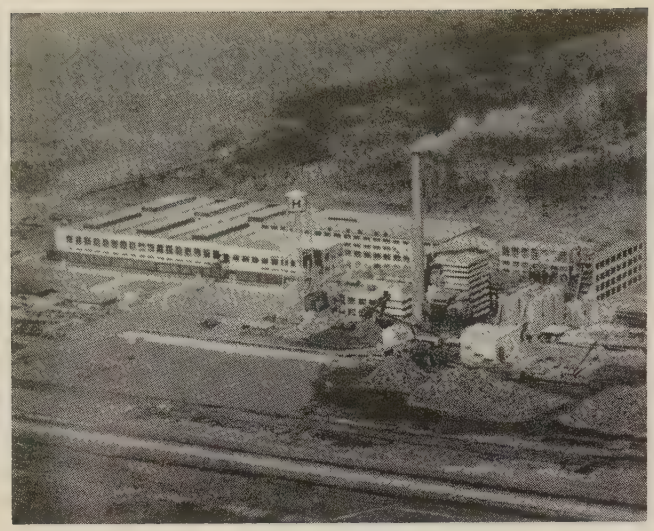
Its vanes force the stock outward, where the impellers rub and refine it against the fixed refining discs. Baled pulp, waste paper, machine broke — even glassine and wet strength — is completely disintegrated in 10 to 40 minutes.

Over 140 sold to date. Ask your Jones representative for details — or write for Bulletin 1019-B.

E.D. Jones **PULP-MASTER**

E. D. JONES and SONS COMPANY • PITTSFIELD, MASSACHUSETTS

BUILDERS OF QUALITY STOCK PREPARATION MACHINERY



Aerial view of the Hudson Pulp & Paper Corp.'s kraft mill at Palatka, Fla.

continuous and batch colored stock systems. A unique arrangement of chests will provide maximum flexibility of preparation of colored stock with a minimum of lost time and production when change-overs are made. These chests in the refining room are also glazed tile, as well as the white water storage chest. Consistence regulators provide a controlled consistency for the screened stock to the hydrating jordans and the two special three-roll heaters. Final stock preparation ahead of the paper machine is furnished by four finishing jordans.

The new paper machine will be a Pusey and Jones 234-inch special fourdrinier MF and MG kraft machine. Stock will be regulated to this machine through a metering stuff box from which the stock will go to a 20,000-g.p.m. fan pump driven by a synchronous motor with an electric coupling for providing speed variations. The fourdrinier is of the latest design and, in addition to the couch roll, has a wire drive roll. There will be a pickup transfer felt and suction roll. The press section consists of a transfer press and a three-roll dual cloverleaf press. The drying section consists of a creping drier and three sections of main driers, with a total of 33 60-inch diameter paper driers and 14 felt driers, including 10 Feeney driers. Between the first and second drier sections there will be a smoothing press and between the second and third sections of driers there is a 12-foot diameter Yankee drier. One 8-roll calender stack, Pope reel, and a high-speed winder will be provided at the finishing end. This machine is designed for a top speed of 2000 f.p.m. and will be driven by an electric sectional drive. The machine is equipped with the usual auxiliaries, including the oiling and condensate systems and vacuum pumps. A new type of tile-lined wire and wet broke pit was designed especially for this new type of machine.

There will be a conventional broke beater at the dry end, supplemented by a broke pulper located in the stock preparation room. All water from the wet broke and wire pit will be pumped to a vacuum saveall for the recovery of fibers in the white water.

A new sextuple effect evaporator unit will supplement the existing unit. The new recovery boiler will be a 250-ton unit equipped with a Cascade evaporator. There is a precipitator for recovering the dust from the gases from the recovery unit.

The existing causticizing plant is being expanded to provide the additional white liquor required. New equipment includes a slaker, causticizing tanks, green liquor clarifier and green liquor dregs washer, lime kiln, and lime mud vacuum filter.

Additional new and reburnt lime storage tanks with lime-handling systems are also included.

Power plant extension consists of one new oil-fired boiler and one new refuse boiler, both duplicates of the existing power boilers. A bark-burning furnace is provided on the refuse boiler. A new 7500-kw. turbine supplies all additional power required.

No expansion to the converting plant is included in this program, as this portion of the mill was doubled recently.

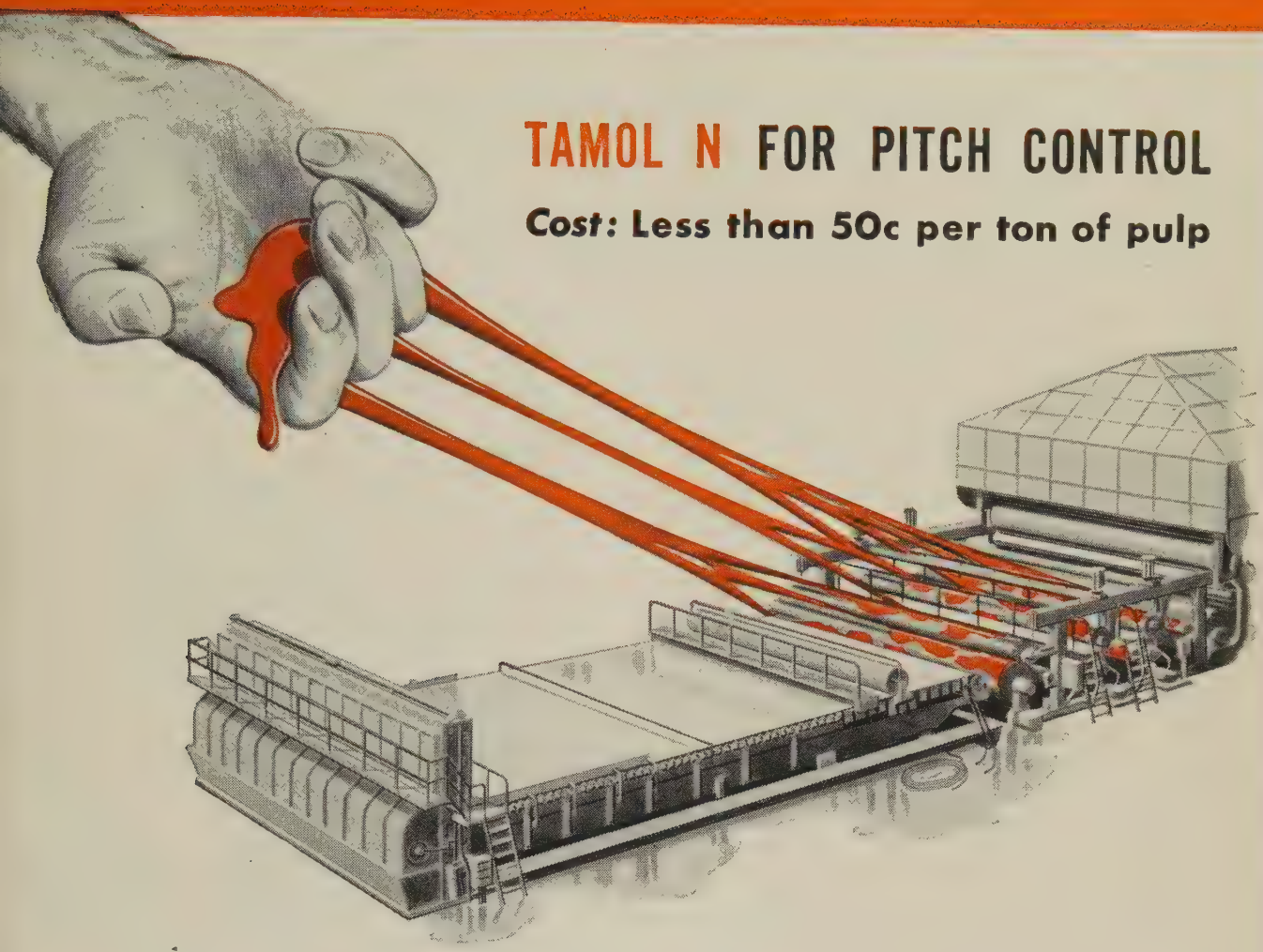
* * *

A new tall oil deodorizing unit incorporating improved design features to give a higher quality product has been successfully put in operation at the Covington, Va., plant of West Virginia Pulp and Paper Co. Designed and erected by the Rust Process Design Co. of Pittsburgh, the new unit has improved the quality of the tall oil product through a higher refinement by means of advanced technique in vacuum distillation. Crude tall oil, composed chiefly of fatty and rosin acids from pinewood, is derived from the black liquor skimmings obtained in the pulping process. Among its refined products are those used in paint, paint driers, various soaps, and numerous other products requiring high qualities of color and odor.

* * *

The Everett Pulp & Paper Co., Everett, Wash., has started issuing a new series of house organs, using the theme "Today It's Different." In the current issue the following condensed catalog of modern presses used in America is given as a convenience for comparing capacity of printing presses in both speed and paper size.

	Maximum sheet	*Printing area*	*Rated speed*
Two-Color Cylinder:			
Harris Envelope P-2 1-2 color	12 1/8 x 20 1/2		10,000
Miller	21 x 28	19 1/4 x 26	4,000
Miehle No. 41 TC	27 1/4 x 41	24 x 38	2,800
Miller	28 x 41	25 x 38	3,500
Miehle No. 46 TC	32 x 45 1/2	29 x 42	2,500
Miehle No. 56 TC	38 x 56	36 1/2 x 52 1/2	2,250
Miehle 6/OTC	46 1/4 x 69 1/2	46 x 65	1,500
One-Color Cylinder:			
ATF Kelly No. 1 Automatic	22 x 28	22 x 28	3,600
ATF Kelly No. 2 Automatic	24 x 35	24 x 35	3,000
Lee No. 38	24 x 36	22 x 35	2,500
Lee No. 42	26 x 40	25 x 38	2,000
Miller Major	28 x 41	25 x 38	4,000
Miehle No. 41 Automatic	28 x 41	24 x 38	3,000
Babcock Optimus No. 6	32 x 45 1/2	29 x 43	2,400
Miehle No. 46.	33 1/4 x 45 1/2	29 x 42	2,800
Babcock Optimus No. 7	35 x 50 1/2	31 x 48	2,000
Babcock Optimus No. 10	39 1/2 x 54	37 x 51	1,850
Miehle No. 56 Automatic	42 x 56	38 x 52 1/2	2,250
Babcock Optimus No. 11	43 x 61 1/2	40 x 59	1,750
Babcock Optimus No. 12	45 1/2 x 64 1/2	42 x 62	1,750
High-Speed Cylinder:			
Multipress Model F (FM)	9 x 12	8 7/8 x 12	6,500
Stoessel Automatic	10 x 12 1/2	9 1/4 x 12 3/4	4,500
Chandler & Price No. 1	11 x 15	11 x 14	4,800
ATF Little Giant No. 5	12 x 18	11 3/4 x 17 1/2	4,000
Chandler & Price No. 2	12 1/4 x 18 1/2	12 x 18	4,800



TAMOL N FOR PITCH CONTROL

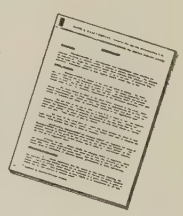
Cost: Less than 50c per ton of pulp

Here's the easy, low-cost way to control pitch, prevent expensive rejects, and eliminate costly shutdowns for cleaning.

Added directly to pulp in the beater, TAMOL N keeps your pitch dispersed in finely divided form. Pitch particles can't clump together to cause spots or holes in paper or to gum the paper machine. And this means fewer rejections and fewer production holdups.

Cost of TAMOL N pitch dispersant is extremely low—usually less than 50c per ton of pulp. Low salt content means added economy—you get what you pay for. If you use alum or clay additions for pitch control, TAMOL will improve their efficiency, too.

Papermakers have known and used TAMOL N for almost 20 years. We'll be glad to send you full details of this time-tested material.



OTHER CHEMICALS FOR THE PAPER INDUSTRY

RHOZYME BB and E-5 Enzymes—for efficient conversion of starch.

UFORMITE Wet-strength Resins—for all furnishes.

TRITON Surface-active Agents—with a score of uses in paper manufacture—in caustic cooking of cotton fiber, as a dispersing agent for pigments and metallic soaps, emulsion-type coatings.

HYDROSULFITE Reducing Agents—for stripping color from rag stock or for bleaching pulp.

HYAMINE Deodorants and Bactericides—for paperboard and glue.

TAMOL, UFORMITE, RHOZYME, TRITON, HYAMINE are trade-marks, Reg. U. S. Pat. Off. and in principal foreign countries.

CHEMICALS FOR INDUSTRY

ROHM & HAAS COMPANY

THE RESINOUS PRODUCTS DIVISION

Washington Square, Philadelphia 5, Pa.

Representatives in principal foreign countries

	Maximum sheet	Printing area	Rated speed
Banthin Cylinder	13 x 20	12 1/4 x 19	3,500
Miehle Verticle V-50	14 x 20	12 1/4 x 19	5,000
ATF Kelly C	17 1/2 x 22 1/2	17 1/4 x 22	4,200
Miller Simplex	20 x 26	19 x 24	4,500
Miller SW	21 x 28	20 1/4 x 28	5,000
Miehle No. 29 Letterpress	22 x 28	20 1/4 x 26	4,500
Rotary:			
Cottrell-Claybourn 5-color	29 x 45	28 1/2 x 44 1/2	5,200
Cottrell-Claybourn 5-color	29 1/4 x 45	28 3/4 x 45	5,200
Cottrell-Claybourn 2-color	29 1/4 x 45	28 3/4 x 44 1/2	5,500
Cottrell-Claybourn 2-color	36 x 48	35 1/2 x 47 1/2	5,200
Cottrell-Claybourn 5-color	36 x 48	35 1/2 x 47 1/2	5,200
Cottrell-Claybourn 2-color	40 x 59	39 1/2 x 58 1/2	5,000
Cottrell-Claybourn 5-color	40 x 59	39 1/2 x 58 1/2	4,500
Miehle 61[a]	42 x 60	41 5/8 x 59	6,000
Cottrell-Claybourn 5-color	43 x 62	39 1/2 x 58 1/2	4,500
Cottrell-Claybourn 2-color	43 x 62	39 1/2 x 58 1/2	5,000
Cottrell-Claybourn 5-color	48 x 71	47 1/2 x 70 1/2	4,200
Cottrell-Claybourn 2-color	48 x 71	47 1/2 x 70 1/2	4,500
Babcock 2-color sheet feed	50 x 72	48 1/2 x 70	4,000
Cottrell 5-color	50 x 72	49 1/2 x 71 1/2	4,000
Miehle 76[a]	52 x 76	51 5/8 x 75	5,500
Platen:			
Chandler & Price New Series 8 x 12	8 1/2 x 11 1/2	8 x 12	3,000
Super Speed Heidelberg	10 1/4 x 15	10 1/4 x 14	5,000
Chandler & Price Craftsman 10 x 15	11 x 15 1/2	10 x 15	3,600
Chandler & Price New Series 10 x 15	11 x 15 1/2	10 x 15	2,700
Kluge Automatic 10 x 15	12 x 15	10 1/4 x 15 1/2	3,500
Colts Armory 2B	12 x 16 3/4	13 3/4 x 19 3/4	2,000
Chandler & Price Craftsman 12 x 18	13 x 19	12 x 18	3,000
Chandler & Price New Series 12 x 18	13 x 19	12 x 18	2,500
Kluge Automatic 12 x 18	14 x 18 1/2	12 x 18	3,000
Laureate	16 x 23 1/4	14 x 22	2,500
Chandler & Price Craftsman 14 1/2 x 22		14 1/2 x 22	2,600
Chandler & Price Craftsman Automatic Unit 14 1/2 x 22	16 x 23 1/4	14 1/2 x 22	2,600
Chandler & Price Heavy Duty Craftsman 14 1/2 x 22	16 x 23 1/4	14 1/2 x 22	2,300
Chandler & Price Super Heavy Duty Craftsman 14 1/2 x 22	16 x 23 1/4	14 1/2 x 22	2,300
Chandler & Price New Series 14 1/2 x 22		14 1/2 x 22	1,800
Colts Armory 5C or 6C	16 x 23 3/4	14 x 22	2,000
Thomson	23 x 33	22 x 32	2,200
Thomson-National Quarto Medium		10 x 15	2,000
Thomson-National Half Medium		13 x 19	1,800

[a] Additional units may be added to print in two or more colors.

The following article on materials handling appeared in the November-December issue of *Link-Belt News:*

Thilmany Pulp & Paper Co., established in 1883 on the Fox River at Kaukauna, Wis., operates a specialty mill.

Here, paper is manufactured and processed from pulpwood to finished product in the form of functional packaging papers for both decoration and protection.

This includes krafts, glassines, greaseproofs; waxed thermoplastic papers; printed and embossed papers; waterproof protective papers; and bags tailor-made to meet the specific needs of the product to be packaged.

Equipped with four rotary 10-foot diam. by 30-foot-high pulp digesters, the plant's sulphate mill has been producing 150 tons of pulp per day, although originally designed for 75 tons daily. The addition of a fifth digester, with new pulp washing and allied equipment, is stepping up the pulp manufacturing capacity to 200 tons a day.

And there are four MG fourdrinier papermaking machines, of 96, 112, and two 132-inch width wire; and three MF fourdriniers of 80, 96, and 132-inch width wire. A new high-speed 132-inch MF machine is currently being installed.

Thilmany also operates a complete converting plant including supercalendering, asphalting, waxing, printing (aniline & rotogravure), and embossing equipment as well as a 30-machine bag factory.

Notable among recent improvements is the very complete modernization of the wood room and the method of delivering wood to it.

The old wood room contained a log deck, two slasher saws, two 10 by 30-foot barking drums, two splitters, two knife barkers, two chippers, one rotary screen, and more than 20 chain conveyors.

P. H. West, the mill superintendent, explained that this wood room "just grew" from time to time until 1928. In 1950 it was considered desirable to start fresh and, in effect, build an entirely new wood-handling system.

The main objectives, of course, were to accomplish greater capacity, cut costs of production, and provide cleaner, more uniform chips for better papermaking.

A particularly noteworthy change is the use of full-length logs of pulpwood. Wood, 8-feet long is now being handled through both the new barker and new chipper, instead of the conventional 4-foot lengths. It is believed that Thilmany is the second mill in the U. S. to bark 8-foot sticks in a barking drum.

This has eliminated wood loss through sawing and by reduction of the number of ends, thus resulting in less oversize "end chips" and less sawdust.

A new "hot pond" measuring 50 feet wide, 250 feet long, 8 feet deep, has put into operation a more economical method of bringing the wood to the wood room.

The 8-foot wood is fed into this pond by a Diesel engine operated, 40-ton capacity, locomotive type crane equipped with 1 1/4-cord grapple, which very efficiently removes the wood from flat cars, holding 20 cords, that were loaded by similar means in the adjacent wood yard.

The plant receives approximately 100,000 cords of pulpwood annually, and uses about 300 cords per day.

About 60% is Canadian jack pine which comes by boat across Lake Superior to Green Bay, Wis., where it is loaded into gondola cars for delivery to Kaukauna. Possibly 30% of the wood pulped is Montana lodgepole pine.

Mr. West explained that the Canadian wood, being slower growing, is denser and contains more fiber per cord than the domestic jack pine, which constitutes 10% of the total and is also inclined to be crooked and poorly trimmed.

Waste water from the evaporators in the chemical recovery plant flows by gravity into the hot pond at 70°F.

This waste water, circulating by means of a pump, carries

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the floating logs to a "jack ladder" located just inside the wood room.

The four-hour soaking period to which the logs are subjected in the hot pond, serves to soften and loosen bark, and remove dirt, snow, and ice.

Consequently, the 8-foot wood is barked dry in the new drum barker, which measures 12 feet diameter by 45 feet long and rotates constantly at 7 r.p.m.

The new chipper is operated by a 600-hp. synchronous motor, measures 88 inch disk diameter, has ten blades, and can handle 8-foot long wood in diameters up to 19 inches.

Knife blades are whirled around at 360 r.p.m. to produce 3600 cuts per minute as the 8-foot wood slides down the funnel-like opening into the chipper, to be cut into $^5/_8$-inch chips about as fast as you can say "Jack Robinson."

With the over-all stepped-up operation in the wood room, now producing better chips and more of them, the digester yield has been increased considerably.

In actual saving, it is estimated that the new wood room will save the company $100,000 annually, including the reduction of shifts from three to two, the wood saving, and the reduction in maintenance.

Above all, this extensive modernization project, involving the expenditure of considerable capital, has placed Thilmany in a much better and more secure competitive position.

We suspect, however, that these savings would be impossible without there also being a very efficient system of conveyors to connect one operation with the next, to make the process rapid and continuous.

The conveyor system was designed and built by Link-Belt Co. at its Pershing Road plant in Chicago, to suit Thilmany's individual needs, and is giving a very good account of itself. The handling capacity is 25 cords per hour.

This L-B conveyor equipment, named in sequence, consists of the following:

1. A quadruple-strand jack ladder No. 1, about 38 feet long, rising from hot pond at an angle of 21 degrees.
2. A horizontal, double-strand feed-in conveyor, about 14 feet long, from head end of jack ladder to drum barker. The 8-foot wood carried on jack ladder crosswise is automatically delivered to this feed conveyor lengthwise. It is planned to convert this to a 20-inch wide belt conveyor in the near future.
3. A 36-inch wide, 57-foot long, flat-roll belt conveyor for receiving the barked or peeled wood as it tumbles out of either of two barking drums. A second new drum may be installed alongside of drum now in service at some future date if required.
4. A quadruple-strand jack ladder No. 2, 23 feet long, rising at an angle of about 43 degrees from belt conveyor (3) and at right angles thereto.
5. A 24-inch wide, 46-foot long, flat-roll belt conveyor for receiving the barked wood lengthwise on an elevated platform and carrying it straight ahead to funnel opening into chipper. This conveyor parallels the other belt conveyor but operates in opposite direction.

There are two men with pike poles straightening and aligning wood in the hot pond, while a third man at foot of jack ladder No. 1 guides the logs into position on conveyor.

Two more men are stationed at foot of jack ladder No. 2, with pickaroons, to inspect and guide the barked wood to the pushers of jack-ladder conveyor.

No attendant is required at funnel opening to chipper. Visitors like to watch the wood spin "down the hatch" to and through the chipper knives.

All three conveyors handling the wood lengthwise, have high sloping steel sides to confine the logs to their intended path.

It is only on the jack ladder conveyors that the wood is handled crosswise.

Both jack ladder conveyors employ four strands of Link Belt Class SM-594 Combination chain with H-1 pushers every fourth pitch.

The barker feed-conveyor uses two strands of C-111B combination chain.

Each conveyor is operated individually from a separate motor.

* * *

A turnout of 41 students in the Curriculum of Pulp and Paper Technology at Western Michigan College met on October 12 to organize a club for future papermakers. A constitution and by-laws were adopted by the group at the initial meeting following the summer recess during which the students held jobs in the paper industry.

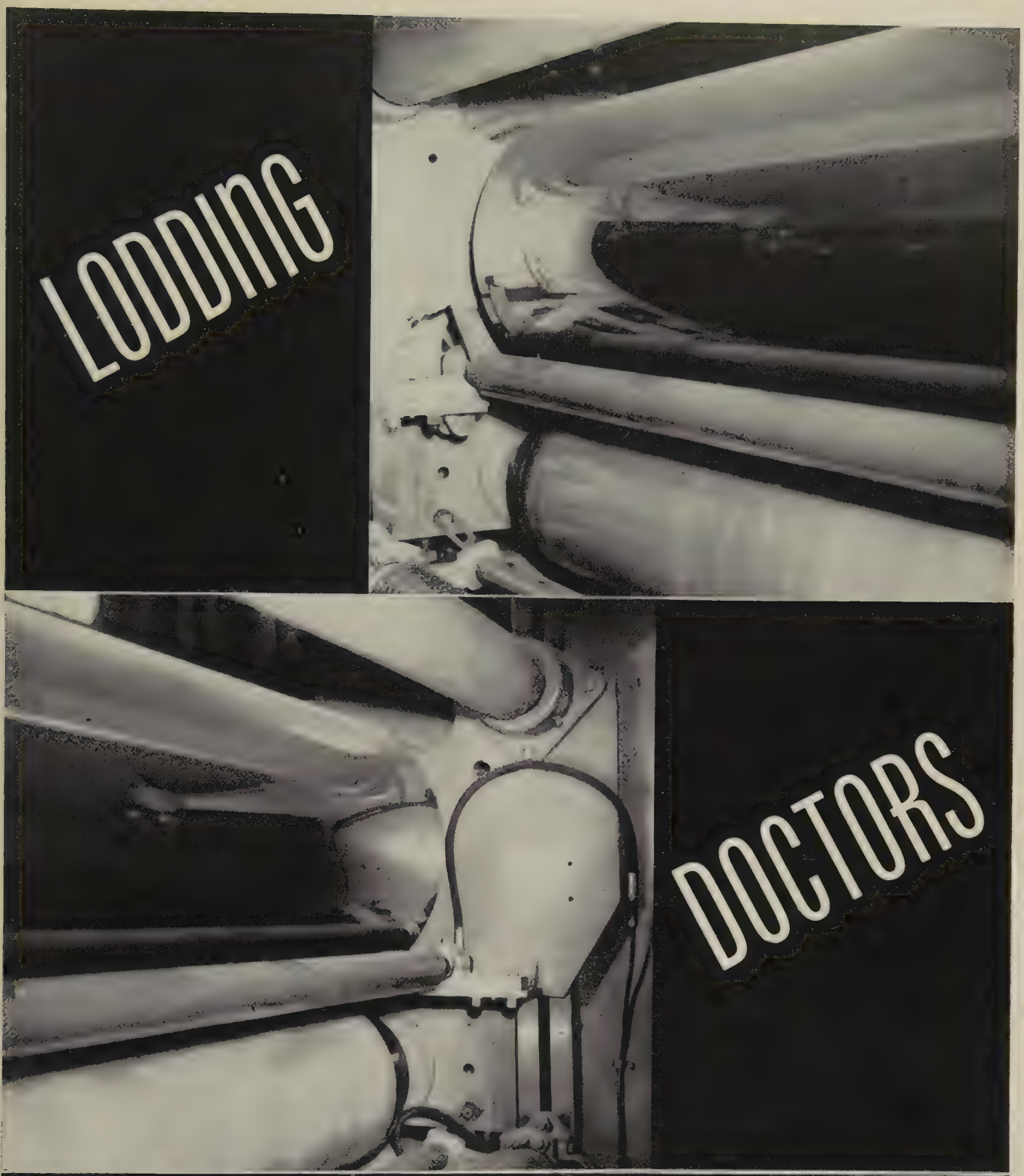
Prior to the election of officers, the group chose the name of "Ts'ai Lun" for their club to honor the first-recorded papermaker in history. Officers were elected as follows: Kenneth L. Maves, President; John F. Triplett, Vice-President; Raymond L. Janes, Secretary; Phillip H. Avery, Treasurer; and Wayne C. Kendrick, Alumni Secretary.

Officers elected by "Ts'ai Lun," newly organized paper makers club at Western Michigan College, are, left to right: Phillip H. Avery, Treasurer; John F. Triplett, Vice-President; Kenneth L. Maves, President; Raymond L. Janes, Secretary; and Wayne C. Kendrick, Alumni Secretary

Active membership will be made up of students in the curriculum of Pulp and Paper Technology, with associate membership open to students taking only a limited number of courses in the paper field. Provision was also made for honorary memberships in the industrial field and for alumni membership. Friends of the college in industry will join the Western Michigan College Faculty as sponsors of the new club.

* * *

Two additional multiroll controlled flow Victory beaters made by The Noble & Wood Machine Co. of Hoosick Falls, N. Y., have been installed in the new kraft pulp and paper mill of the Hudson Pulp and Paper Corp. at Palatka, Fla. The two new triplex units are duplicates of the three-roll Victory beater which was installed in Hudson's Palatka mill in August, 1949. Officials of the Florida firm state that the original Victory beater has been in continuous operation since installation with no shutdowns caused by mechanical difficulties, and that the original bedplate and roll bars still in the beater show only a small amount of wear. The unique design of the Noble & Wood Victory beater makes possible hydraulically controlled bedplate-to-roll pressures of up to 40 tons, continuous stock flow of up to 1000 g.p.m., and continuous daily production of 200 or more tons for each triplex unit. In addition to the new Victory beaters, the Hudson Pulp



Reflecting the quality of
Lodding Oscillating Supercalender Doctors

Lodding Engineering Corporation, Worcester, Massachusetts

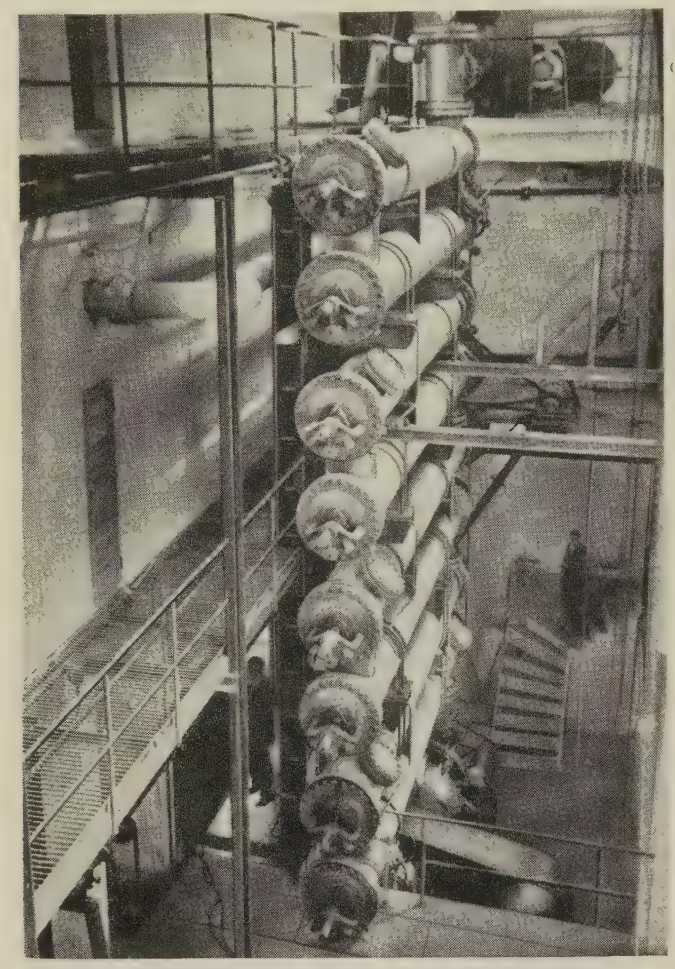
Represented by

W. E. Greene Corporation, Woolworth Building, New York

and Paper Corp. has also included four Noble & Wood Unifiners and nineteen Noble & Wood propeller agitators.

* * *

The Chemi-pulper unit at Veitsiluoto, Finland, was installed and started up this past Summer and it is to operate primarily on waste spruce and pine which is available from the Veitsiluoto sawmill operation. Paper and Industrial Appliances, Inc. supplied the Chemi-pulper continuous digester, which operates with an Asplund defibrator, and some of the auxiliary equipment for this pulp mill. R. G. Goodwin, Director Sales Engineering for the New York firm, was present for the start-up of the first Finnish Chemi-pulper installation.



View of the Chemi-pulper installed at Veitsiluoto, Finland

Shown in the photograph are the key personnel at the Veitsiluoto plant standing near the base of the Chemi-pulper and reading from left to right:

Junno Laanti, Assistant to Chief Chemist
Matti Rautakorpi, Planning Engineer
Heikki Sihtola, Mill Manager
R. G. Goodwin, Paper & Industrial Appliances, Inc.
Seppo M. Saukkonen, Chief Chemist and Semichemical Pulp Mill Superintendent

The complete Chemi-pulper installation, including the Asplund defibrator which is located in a pit, is clearly shown in the other picture.

The general flow at the Veitsiluoto operation calls for continuously feeding the screened pine or spruce chips to the 8-pipe Chemi-pulper, and equipment is available to presoak the chips if required. The hot stock coming from the Chemi-pulper is refined in a Sprout-Waldron refiner. The pulp passes on to the hot stock chest and is pumped directly to a stainless steel vacuum washer which is equipped with a repulper. The pulp may go directly to a Kamyr wet machine from the washer or it can pass over a screen and thickener and then to the wet machine.

Junno Laanti, Matti Rautakorpi, Heikki Sihtola, R. G. Goodwin, and Seppo M. Saukkonen

Most of the equipment in the Veitsiluoto continuous pulp mill is made from 316 stainless steel; therefore, they are in a position to use either acid or alkaline cooking liquors.

The pulps produced are in the 50 to 75% yield range, using either acid or alkaline cooking liquors.

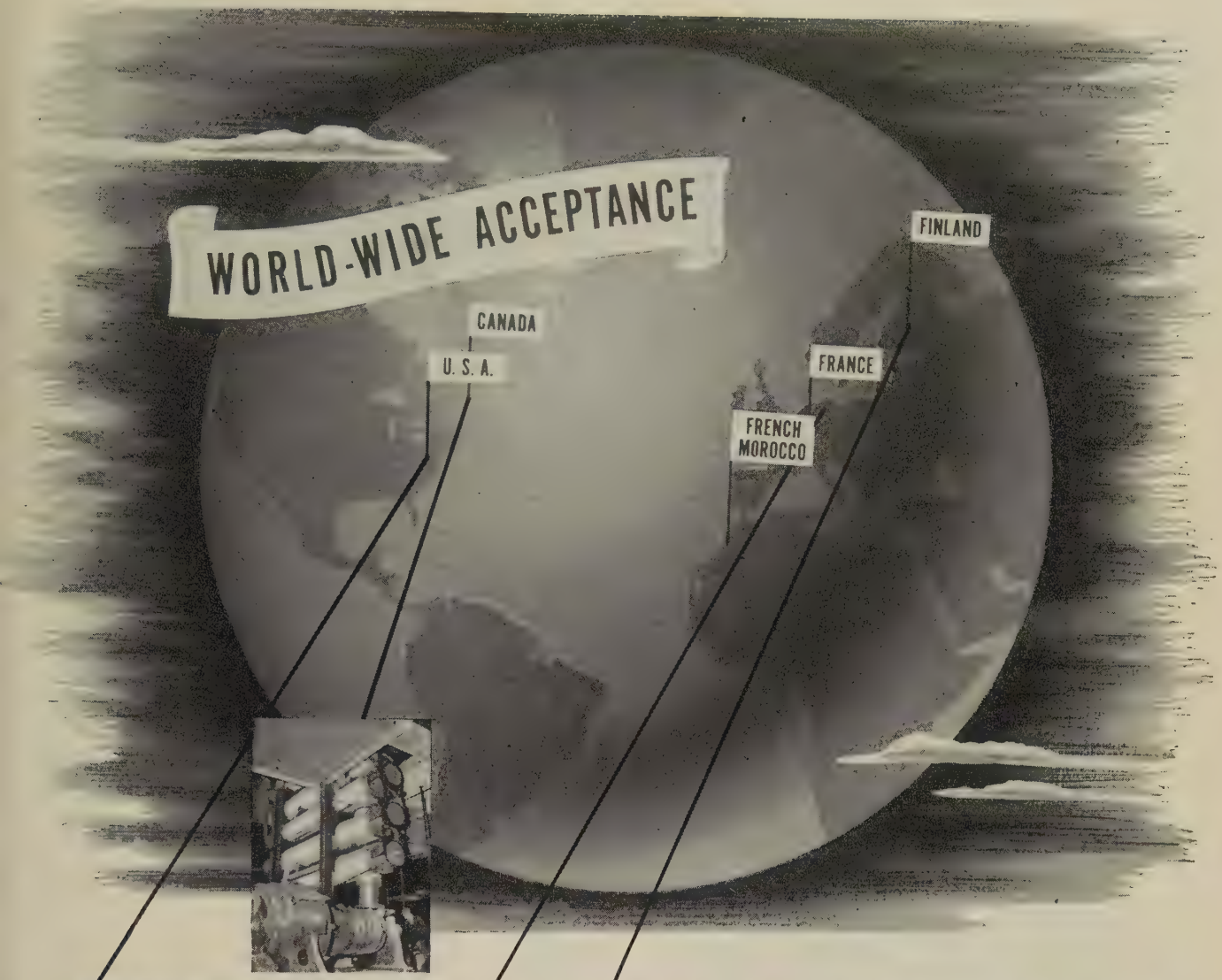
According to the latest information received by R. G. Goodwin who has recently returned from an extended European trip, the Veitsiluoto Chemi-pulper at the present time is operating with waste spruce and an alkaline cooking liquor producing pulp for export.

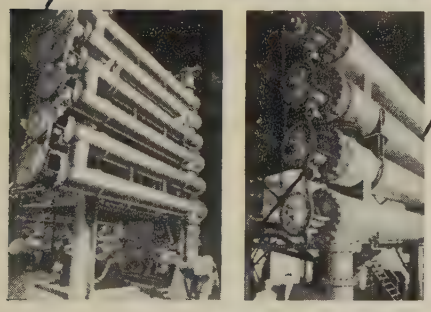
* * *

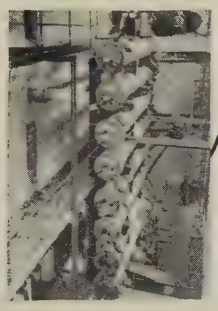
A new and fourth edition of "The Why and How of Business Paper Advertising" has been announced by Stephen Goerl Associates, New York advertising agency, which published the original edition in 1948. The first three editions consisted of nearly 20,000 copies and were bought by advertising agencies, advertisers, and publications at a nominal price. The concise 32-page pocket-size booklet points up the value of advertising in business papers, and contains practical suggestions for using such advertising more effectively. Claiming no complete coverage of the subject, the booklet stresses the importance of field research and quotes the 16 suggestions put forth by the Professional Development Committee of the National Industrial Advertisers Association. Campaign objectives, copy, layout, artwork, color, and other basic subjects are covered. The chapter dealing with institutional and public relations advertising is particularly timely today, in view of the material shortages in many industrial fields, and the shifting of emphasis from immediate sales objectives to future good will. Stephen Goerl, the author, gives ten basic rules of good business paper advertising which he says are not a "cure-all" but which are easy to follow and should serve as a valuable guide to many advertisers in determining whether their present advertising is on the right track. Copies of "The Why and How of Business Paper Advertising" are available at 25¢ from Stephen Goerl Associates, Inc., 48 E. 43rd St., New York 17, N. Y.

* * *

The Sulphur Committee of the International Materials Conference reports that the expected world sulphur production for 1952 will be 6,255,600 tons against requirements of







PULP DEFIBERED IN ASPLUND DEFIBRATOR

CHEMI-PULPER®
continuous digester

The ever-increasing, world-wide use of the CHEMI-PULPER continuous digester has mill-proved the value of its principle of continuous cooking under controlled conditions. More and more installations are being made in mills using either hard or soft woods to *economically* make pulps of predetermined quality and yield to meet specific requirements. Such pulps are produced for making .009 for corrugating; flooring, roofing and automotive felts, and insulating board. Effects substantial savings in steam, power and labor costs compared with conventional pulping methods. Bulletin C2 gives the facts . . . yours for the asking.

MANUFACTURED AND SOLD IN CANADA BY ALEXANDER FLECK LIMITED, OTTAWA, ONTARIO.

PAPER and INDUSTRIAL APPLIANCES, INC.
122 EAST 42 nd STREET ● NEW YORK 17, N. Y.

7,596,600 tons. Pyrite production is expected to be 4,745,600 tons against requirements of 5,000,300 tons. Due to expanded use of sulphuric acid, sulphuric acid, sulphur dioxide and carbon bisulphide, together with increased consumption of sulphur derivatives by agriculture and other industries, the demand for sulphur in 1952 is expected to show an increase of 40% over the 1949 figures.

* * *

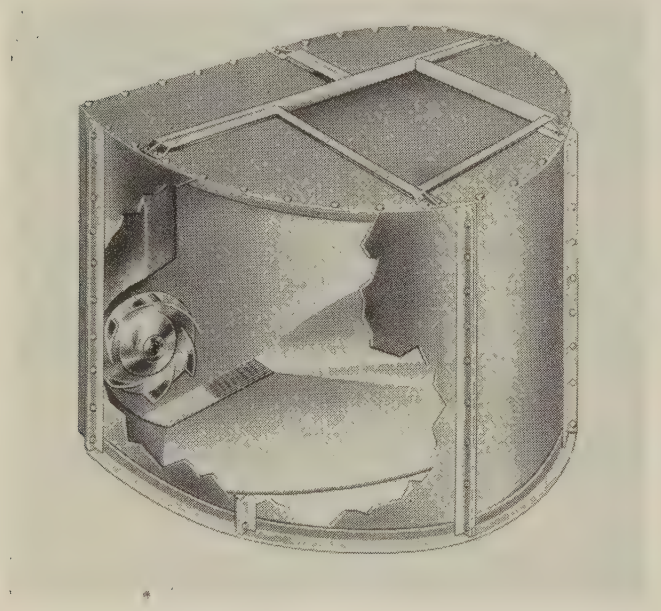
Hamilton Laboratories, Inc., has been formed as a Maryland corporation at Wood Ridge, Md., and has purchased the patents of the Hamilton Laboratories of Asheville, N. C., which has been dissolved. R. T. Vanderbilt Co., Inc., of New York City will represent the new firm exclusively for the sale of all of the organo-mercurial products formerly offered by the Asheville concern under the trade-mark Merphenylopes, to the pulp and paper, and paper converting industries of the United States and Canada.

* * *

Frank W. Egan & Co. has added a manufacturing plant to its Bound Brook, N. J., facilities. The new property is adjacent to its present building on Lincoln Blvd. The company manufactures machines and equipment for the paper converting and plastics industries.

* * *

A new bulletin describing its side-drive pulper, now known as the Aquapulper, has just been released by the Cowles Co., Inc., of Cayuga, N. Y. The bulletin explains in detail how hard stock can be defibered quickly and completely using



Cowles Aquapulper for pulping and defibering hard stock

the company's Aquapulper. The unit, it points out, feature a side-drive design that permits the use of small, high-speed impellers which are unusually efficient for pulping and defibering. The extraction chamber has been redesigned to give a flat bottom on the Aquapulper which permits installation directly on the floor with no excavation necessary. The Aquapulper outlet can be located either in the bottom or the side of the tub to permit easy piping for the stock discharge.

* * *

Downingtown Mfg. Co., Downingtown, Pa., has been licensed to build the Masson-Scott high-speed cutters and layboys in the U.S.A. One unit is in operation and two others are being built. Masson-Scott is a British machinery firm.

* * *

Hercules Powder Co., Paper Makers Chemical Dept., has opened a new unit in Savannah, Ga., for the production of Kymene 138 and 234, wet-strength resins for paper. Kymene resins are made also at Mansfield, Mass., and are used in making toweling, map paper, V-board, blue print paper, and paper bags.

* * *

H. Wyatt Johnston resigned as Vice-President of Sutherland Refiner Ltd. on Januray 1. He will act as consultant to the Sutherland interests in Canada, United States, and foreign countries. He will also be a consultant to the pulp and paper industry generally and will be located at 4048 Gage Road, Montreal, P. Q.

* * *

Eriez Manufacturing Co., Erie, Pa., has issued a new bulletin (No. 561) on Magnetic Separators for Pulp, Paper, and Felt Mills.

* * *

Morden Machines Co. has announced the appointment of the Orton Corp., Fitchburg, Mass., as its Eastern sales representative for the Morden Slush-Maker and Stock-Maker.

* * *

All sales activities of the Du Pont Co.'s Organic Chemicals Dept. are now under the direction of Douglas C. Newman. Miles A. Dahlen succeeds Mr. Newman as Director of Sales for the Dyestuffs Division.

The name of the Du Pont Co.'s Rayon Dept. has been changed to Textile Fibers Dept. and, in addition to rayon will handle Orlon (acrylic fiber) and Dacron (polyester fiber). Robert L. Richards is General Manager of the department. Andrew E. Birchaven, Jr., is assistant General Manager.

* * *

The 7th National Chemical Exposition, sponsored by the Chicago Section of the A.C.S., will be held at the Chicago Coliseum on Sept. 9–13, 1952.

* * *

E. A. Yaeger has been appointed Vice-President of National Distillers Chemical Corp., 120 Broadway, N. Y. The company manufactures sodium peroxide.

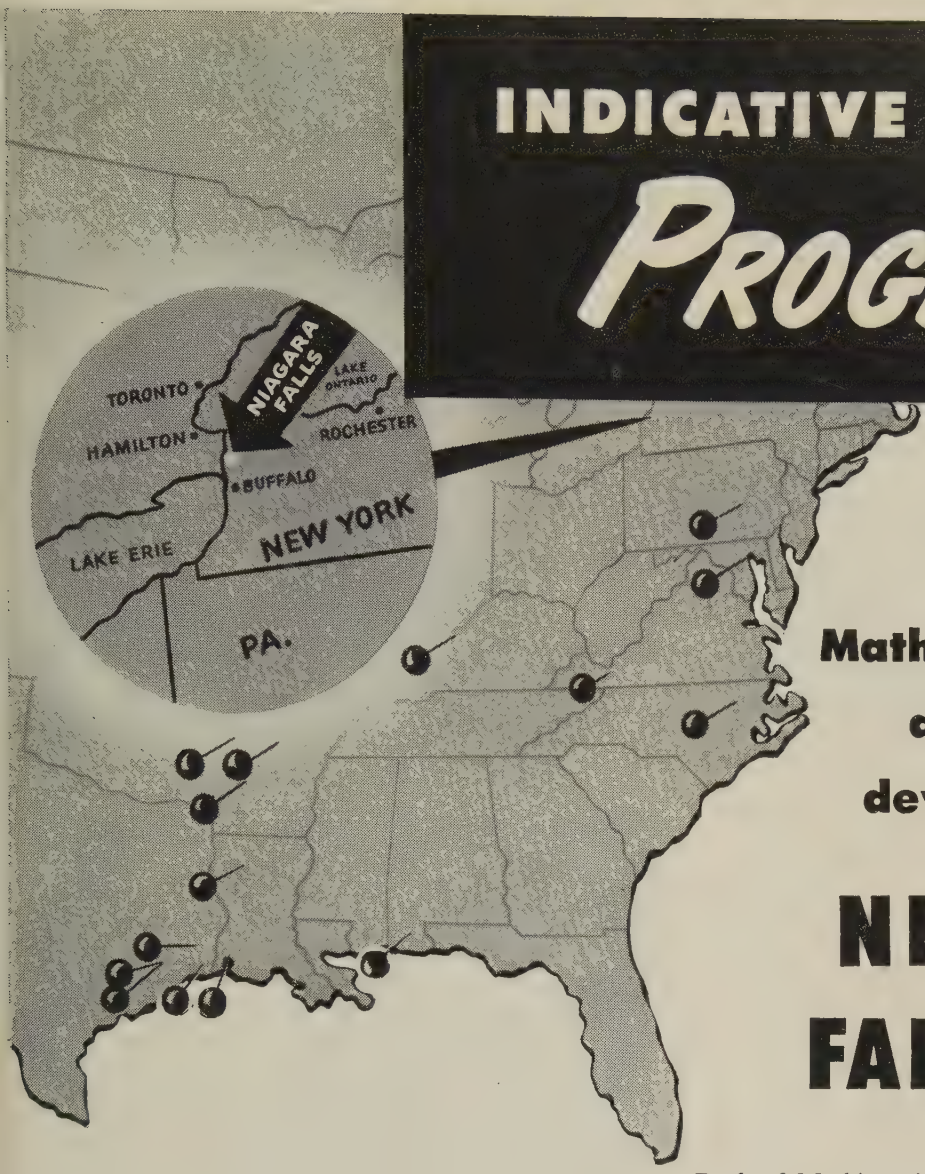
* * *

American Resinous Chemical Corp. has doubled its production through recent plant expansion. The company's products are called Arwaxes. These are concentrates of synthetic rubbers and resins in paraffin and microcrystalline wax and are used to improve adhesion, flexibility, heat-seal, antiblocking and gloss properties of the waxes. Butyl rubber, Vistanex, S. Polymer, and Polyethylene Arwaxes are now being produced. Vistanex Arwax concentrates are used in processing cheese wrappers and for laminating glassine to neutral kraft to make greaseproof barrier wraps. They are also used with asphalt to make improved waterproof paper. Butyl Arwaxes are used for laminating glassine to glassine, glassine to boxboard and kraft to kraft both plain and creped. They are also used to improve the heat-seal strength and water vapor transmission resistance of paraffin wax glassine coatings. S-Polymer Arwax Concentrates make tough hot melt coatings used in fabricating waterproof and water-vaporproof papers and heat seal coating for glassine and paper. Polyethylene Arwaxes are used in making locker papers, bread wrappers, box overwraps, and folding boxes for frozen foods.

* * *

Hooker Electrochemical Co. plans to build a new 10 million

INDICATIVE OF PROGRESS...



Mathieson research and product development at

NIAGARA FALLS, N.Y.

Back of Mathieson's expanding service to American industry is a well-integrated program of research and product development. The growth and diversification of output at Mathieson's Niagara Falls plant, for example, is the result of skilled work in this field. First production, over 50 years ago, consisted only of caustic soda and bleaching powder. Today, stemming from the original electrolytic process, Mathieson is producing these essential chemicals:

Caustic Soda • Chlorine • Ammonia • HTH Hypochlorite Products • Sodium Chlorite Products • Sodium Methylate • Hydrazine Products • Benzene Hexachloride

Mathieson's research and development program has been expanded to all 16 strategically situated plants. Out of this program are coming many chemicals which may fit into your future production and marketing plans. Mathieson Chemical Corporation, Mathieson Building, Baltimore 3, Maryland.

MATHIESON HEAVY CHEMICALS

Sulphuric Acid • Processed Sulphur • Soda Ash
Caustic Soda • Bicarbonate of Soda • Ammonia
Sodium Chlorite • Nitrate of Soda • Chlorine
Calcium Hypochlorite

Mathieson CHEMICALS

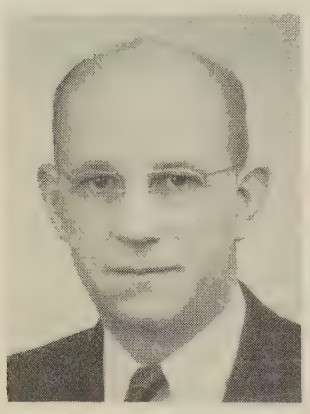
9329

SERVING INDUSTRY, AGRICULTURE AND PUBLIC HEALTH

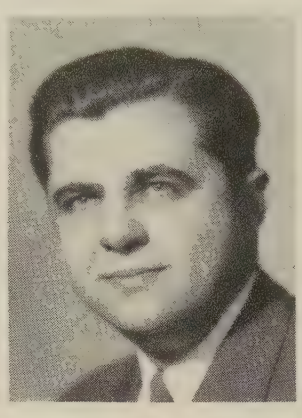
dollar plant for the production of chlorine and caustic soda at Montague, Mich. The plant will be in operation in 1953.

* * *

Otto W. Hein has been elected Vice-President of the Black-Clawson Co., Hamilton, Ohio. He will also be General Manager of the Shartle Brothers Machine Co. Division. Mr. Hein was formerly Director of Research and Development of the B-C, Shartle and Dilts organizations.



Otto W. Hein, The Black-Clawson Co.



A. G. Dreis, P. H. Glatfelter Co.

* * *

Arthur G. Dries is now Director of the Product Improvement Department of the P. H. Glatfelter Co., Spring Grove, Pa.

* * *

The San Joachim Div., Fibreboard Products, Inc., at Antioch, Calif., has installed a General Electric Co. beta-ray thickness gage on its paper machine.

* * *

Raybestos-Manhattan, Inc., Manhattan Rubber Division, Passaic, N. J., has recently issued bulletins No. 6868E on Condor V-belts and No. 6951 on the Ray-Man "F" Conveyor Belt.

* * *

Bleaching of neutral sulphite semichemical pulp is discussed in the October, 1951, issue of *Better Fibers* issued by Sprout, Waldron & Co., Muncy, Pa.

* * *

Engineering Products Co., Inc., 122 S. Michigan Ave., Chicago 3, Ill., has just issued a new catalog about its trackage systems for moving heavy loads inside the plant.

* * *

The Chicago International Trade Fair will be held at the Navy Pier, Chicago, Ill., on March 22 to April 6, 1952.

* * *

Frank H. Jewett, formerly of S. D. Warren Co., is now New England District Representative of Gallowhur Chemical Corp. of New York.

* * *

Clinton K. Royce who recently resigned as Head of the Packaging Section of the Department of the Navy's office of Naval Material has joined the staff of Vanant Products, Milwaukee, Wis., manufacturers of packaging materials. Mr. Royce has been a speaker on recent TAPPI Local Section programs.

John S. Bates, who has been Manager of the Montreal office of Price & Pierce, Ltd. since 1940, is retiring from active management of the office, but will remain with the company as a consultant. E. G. Kirby will succeed Mr. Bates as Montreal manager.

* * *

Alfred Lippman, Jr., has been appointed Manager of Laboratories for the Commonwealth Engineering Co., Dayton, Ohio.

* * *

The Dorr Co., Barry Place, Stamford, Conn., has issued a 28-page bulletin (No. 3001) on Dorr Thickeners. The company now manufactures more than 30 types of sedimentation units of the unit compartment and tray types.

OBITUARIES

Gunnar Frederik Magnuson

Gunnar F. Magnuson, Managing Director of A/B Defibrator, Stockholm, Sweden, died at Stockholm on Saturday, Nov. 24, 1951.

Gunnar F. Magnuson

Mr. Magnuson was born at Sandviken, Sweden, on Nov. 3, 1884, and graduated from the Technological Institute of Stockholm in 1907. He was well known to a large number of individuals in the United States and Canada.

Since 1907 he had a long and illustrious career in the pulp and paper industry as engineer, superintendent, and managing director of pulp and paper companies in Sweden. He was President of the Swedish Paper and Pulp Engineers Association from 1929 to 1937 and was a member of TAPPI since 1923.

George L. Welp

George L. Welp, 61, nationally known authority on color in advertising, died on Nov. 29, 1951, in Long Island College Hospital after a five-week illness.

Mr. Welp, who lived in Great Neck, was advertising and sales promotion manager for the Interchemical Corp.'s printing ink division. He had been with the firm since 1933.

Born in Brooklyn, he worked with what is now the Compton Advertising Agency until 1930. He operated his own agency before joining Interchemical.

He was co-author and designer of the International Printing Ink Corp.'s "Three Monographs on Color," now considered a basic textbook in the color printing field.

For many years he was a member of the Packaging Council of the American Management Assn. and represented his firm

Here's a man you ought to know!

—He's A Cyanamid Technical Service Expert
His Experience, Skill, Judgment Are All "On Paper"

Here's a man whose know-how is all on paper, the application of chemicals to paper—just where it has to be if he's to serve you competently.

From one end of the paper mill to the other, from beater to finished sheet, he knows every inch of the route travelled by paper. He knows most of the production kinks likely to occur along the way. And he knows how to meet them.

Best of all, he knows the chemistry of paper . . . and he knows just what chemicals in the extensive Cyanamid line will give best results in your processes.

Call him in if you have any questions about the newest Cyanamid developments—or if you just want to get the best possible results from the Cyanamid chemicals you're now using.

ALWAX* Sizes • WAXINE® Sizes • Rosin Size • PAREZ® Resins • Synthetic Resins • Casein • Sulfonated Oils • Filters Defoamers • Soda Ash • Caustic Soda • Salt Cake • Acids • Clays AEROSOL® Wetting Agents • CALMICRO® Calcium Carbonate AZITE® 900 Liquifier • Aluminum Sulfate • Sodium Phospha Aluminate.

*Trade Mark

Sales Offices: Boston • Philadelphia • Pittsburgh • Baltimore Charlotte • Cleveland • Cincinnati • Chicago • Detroit Kalmazoo • St. Louis • Los Angeles • San Francisco • Seattle In Canada: North American Cyanamid Limited, Toronto and Montreal

AMERICAN Cyanamid COMPANY
INDUSTRIAL CHEMICALS DIVISION, DEPT. T-1
30 ROCKEFELLER PLAZA, NEW YORK 20, N. Y.

on the Intersociety Color Council and as official representative in the Technical Association of the Pulp and Paper Industry.

William E. Corbin

William E. Corbin, 82, well-known retired Brown Co. executive, died suddenly at his home in Cascade on Saturday, Oct. 13, 1951. At the time of Mr. Corbin's retirement in 1933, he was Superintendent of both the Cascade and Riverside Paper Mills of Brown Co.

Mr. Corbin was born in Charlestown, N. H., on March 15, 1869. At the age of 16 he started working in the paper industry in a newsprint mill owned by Ex-Governor Cheney at Manchester, N. H.

William E. Corbin

Mr. Corbin went to Berlin in May, 1892, to help start the first paper machines of the Riverside Paper Mill of the Berlin Mills Co. (now Brown Co.). He became superintendent of the mill in Oct., 1892, and some time before 1904, he was made superintendent of the Groundwood Mill in addition to the Paper Mill. He loved to tell that in the early days there were only five men in the entire mill—three men on the paper machine and two men, himself and the purchasing agent, for overhead. No instruments were used for testing the newsprint; Mr. Corbin tested all the paper himself by forcing his thumb through it.

In 1903, Mr. Corbin supervised the preparation of the specifications for the Cascade Paper Mill and the new paper machines. In 1910, Mr. Corbin started production of kraft paper at the Riverside Paper Mill; very little kraft paper was manufactured in the United States at that time.

Mr. Corbin was commonly referred to as the "Father of Brown Co. paper towels." The trade name for Brown Co. paper towels—NIBROC—is Corbin spelled backwards. Mr. Corbin pioneered in the manufacture of a number of kraft papers. Among these were papers for cement bags, onion bags, electrical cables, gumming, and bleached kraft paper for coffee bags. He was a former mayor of Berlin, N. H., and resided in that city for 60 years. He is survived by his wife, Mrs. Mary Richardson Corbin; one daughter, Mrs. Roy E. Brawn of Berlin; one grandson, William Corbin of Arlington Mass.; and one great-granddaughter.

RECENT BOOKS

Pulp and Paper Manufacture. Vol. 2. Edited by J. Newall Stephenson. McGraw-Hill Book Co., New York, 1951. Cloth, 6 × 9, 587 pages. $7.50.

This is the second volume in the new series of textbooks prepared under the direction of the Joint Textbook Committee of the United States and Canada. The new series of four volumes replaces the famous five-volume series "The Manufacture of Pulp and Paper" which has served the industry since 1921.

The supplementary title of Volume 2 is "Preparation of Stock for Paper Making." A large number of members of TAPPI and the Technical Section of the C.P.P.A. serve well as authors of the various chapters. J. N. Stephenson of *Pulp and Paper of Canada* continues his excellent work as Editor of the entire text.

Since most individuals in the industry are familiar with the old series the general nature of this series of volumes is self-evident. A review of the main chapter titles will indicate the scope of the present volume: (1) Pulping of Rag and Other Fibers; (2) Waste Papers; Board-Stock Preparation; Deinking Process; (3) Beating and Refining; (4) Fillers and Loading; (5) Sizing of Paper; (6) Paper Coloring; and (7) Semichemical, High Yield Chemical, and Special Groundwood Pulping Processes.

Although Chapter 7 rightfully belonged in Volume 1 it is just as well that it appears in the second volume since Volume 1 was an extremely large book and could be well relieved of this chapter. Furthermore the extensive stock-preparation aspect of semichemical pulping is emphasized by its presence in Volume 2.

The many authors of the chapters of the four volumes and the even larger number of reviewers merit a very large amount of appreciation on the part of the thousands of individuals who will look upon these books as their bibles of pulp and papermaking for several years to come.

Dictionary of Paper. 2nd Ed. American Paper & Pulp Association. New York, 1951. Cloth, 6 × 9, 393 pages. $6.50.

The first edition of the Dictionary appeared in 1940 and became exhausted in 1950. In view of the many advances made in the industry it was decided to revise it completely and issue a second edition. Many individuals in the industry representing its manufacturing, sales, and technical departments carefully scrutinized the first edition and made sure that the definitions to appear in the present volume represented the current understanding of the industry.

In addition to thousands of paper definitions the volume contains classifications and definitions of pulps and classification of waste materials used in the paper and board industries.

Statistical Methods for Chemists. By W. J. Youden, National Bureau of Standards. John Wiley & Sons, Inc., New York. Cloth, 6 × 9, 126 pages. $3.00.

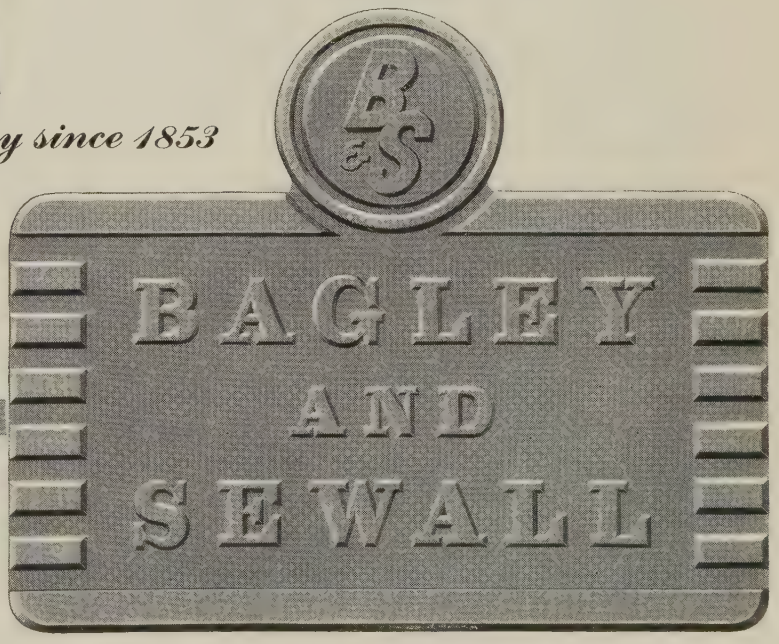
With the growing interest in the use of statistical methods in the paper industry there is a need for simple and elementary volumes that will provide a knowledge of the techniques involved. As stated by the author in the

We can help on those –

PAPERMAKING PROBLEMS...

Bagley and Sewall's constant research and development, not only in theory but in the direct application of newly designed machinery to papermaking problems has resulted in the most practical and economical machinery designs for present day competitive paper mill operation . . .We invite paper mill inquires to our engineering staff for information and survey applications of the latest Bagley & Sewall papermaking machinery developments for either mill modernization or expansion programs.

designers and builders of papermaking machinery since 1853



WATERTOWN • NEW YORK

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Finland Representative
Aktiebolaget Ekstroms Maskinaffar
Helsingfors, Finland

Foreign Representative
Castle and Overton, Inc.
630 Fifth Ave., Rockefeller Center, New York 20, N.Y.

preface "This book is written for those who make measurements and interpret experiments. The book is characterized by an absence of statistical theory and proofs. I am convinced, from observation, that young men who have been given a fine mathematical grounding in statistics often have an inadequate grasp of the ways in which measurements behave. Observation has shown equally that most experimental workers have a very real understanding of the meaning of their measurements. The purpose of this book is to make available to the scientist the modern statistical system of units for expressing scientific conclusions." Dr. Youden has, for twenty years or more, been a leader in both the chemical and statistical fields.

Industrial Water Pollution. By Marvin D. Weiss, R. S. Aries & Associates. Chemonomics, Inc., New York, 1951. Paper-bound, 6 × 9, 142 pages. Photo-offset. $5.00.

This book is a digest of state water pollution control legislation. The provisions of the Federal Water Pollution Control Act of 1948 are summarized. The essential provisions of the laws of each state having specific statutes on stream pollution are reviewed, giving the date of passage of the acts, the composition of the control agency, its authority, requirements, and enforcing procedure. The classification of waters and standards of water quality adopted by a state are given in their entirety. References are made to interstate agencies. These are tabulated in an appendix. The publication is of current interest and value to industry and others engaged in water pollution control.

Styrene. By A. L. Ward and W. J. Roberts, Pennsylvania Industrial Chemical Corp., Chester, Pa. Interscience Publishers, Inc., New York, 1951. Paper-bound, $6^1/_2 \times 9^1/_4$, 126 pages. Varityped photo-offset. $3.50.

This booklet is an addition to the series on Monomers, a collection of data and procedures on the basic materials for the synthesis of fibers, plastics, and rubbers, edited by E. R. Blout and H. Mark. About 200 references are quoted in describing the preparation, purification, care and handling, physical and thermodynamic properties, and polymerization of styrene. The publication is of value to research chemists in the paper and coating industries interested in new packaging paper development.

Chemistry of Wood. By Erik Hagglund, Swedish Forest Products Research Laboratory, Stockholm, Sweden. Academic Press, Inc., New York, 1951. Cloth, 6 × 9, 631 pages. $13.50.

This book is a new revised English edition of "Holzchemie," the first edition of which was published in 1928. Since the second German edition was published in 1939 great studies have been made in the field of wood chemistry. Special attention is paid to the chemistry of cellulose and lignin. In preparing special chapters the author was ably assisted by such outstanding research men as E. Adler, H. W. Giertz, L. Enebo, H. Erdtman, T. Enquist, S. E. Hagglund, and P. W. Lange.

The following chapter titles indicate the nature of the book's contents: Wood Tissue; Physical Properties of Wood; Wood Components and Their Chemical Properties; Pulping of Wood (Acid and Alkaline) Semichemical Pulping; Deliquification with Bleaching Agents; Chemical Processes in the Carbonization of Wood; Behavior of Wood in Storage; and the Natural Decomposition of Wood.

Chemical Formulary. Vol. IX. By H. Bennett. Chemical Publishing Co., Brooklyn, N. Y., 1951. Cloth, $5^1/_2 \times 8^1/_2$, 648 pages. $7.00.

There is an old rule to the effect that nothing should be thrown away by a research laboratory and every scrap of information that may possibly be of future use should be filed. In Volume 9 and the eight preceding volumes Mr. Bennett has endeavored to provide the latter service. It is quite likely that hundreds of research men have thumbed through these volumes to locate an idea; glass stoppers may be stuck—how to loosen them; what surface active agent to use; how to mix a special paint, etc. Odds and ends to be sure; but very useful when needed.

Punched Cards. By Robert S. Casey, W. A. Schaeffer Pen Co., and James W. Perry, Mass. Inst. Technology. Reinhold Publishing Co., New York, 1951. Cloth, 6 × 9, 506 pages. Illustrated. $10.

There is a growing awareness to the need of doing something about the tremendous mass of information that is being published and coming out of every type of research laboratory. The librarians are battling the problem and have done a good job in indexing. The microfilm and other methods have contributed toward the solution of the problem. More and more attention is being paid in business and scientific fields to machine tabulation and the handling of vast amounts of data through the medium of punched cards. Several paper companies have adopted punched card techniques to assist them in accounting procedures: punched cards cannot make complex problems simple but they can make them less complex and some simple problems less tedious and time consuming.

The author deals with manipulation of hand-sorted cards, simple coding procedures, commercially available systems, report indexing, identification of unknown substances, patent searching, production control, correlation of research data, literature searching, classification systems. A bibliography on the uses of punched cards is appended.

The Development of American Industries. 3rd Ed. By J. G. Glover and W. B. Cornell, New York University. Prentice-Hall Co., New York, 1951. Cloth, 6 × $9^1/_4$, 120 pages. $8.00.

This interesting volume covers the economic significance of 41 major industries, including pulp and paper, lumber, textiles, chemicals, newspaper, and others. The section on the Pulp and Paper Industry was prepared by Louis T. Stevenson, Economist for the American Paper & Pulp Association. Twenty-three pages are devoted to this industry in which the author briefly covers its history, raw materials, processes, structure, financial background, and distribution. It is a useful reference to individuals and companies having an interest marketwise in the major industries.

Microbial Decomposition of Cellulose. By Ralph G. H. Siu, Research Director, U. S. Army Quartermaster Corps. Reinhold Publishing Corp., New York, 1951. Cloth, 6 × $9^1/_4$, 531 pages. $10.

With world-wide shipment of packages again a matter of concern the present book meets a need. Such a book was needed early in World War II but did not exist. It is an extensive compilation of cellulose-destroying fungi, bacteria, actinomycetes, and their activities. Practical methods of mildew-proofing are given as well as a thorough discussion of theoretical and practical factors in test methods for mildew resistance. Although the major emphasis is placed upon cotton textiles the facts are appli-

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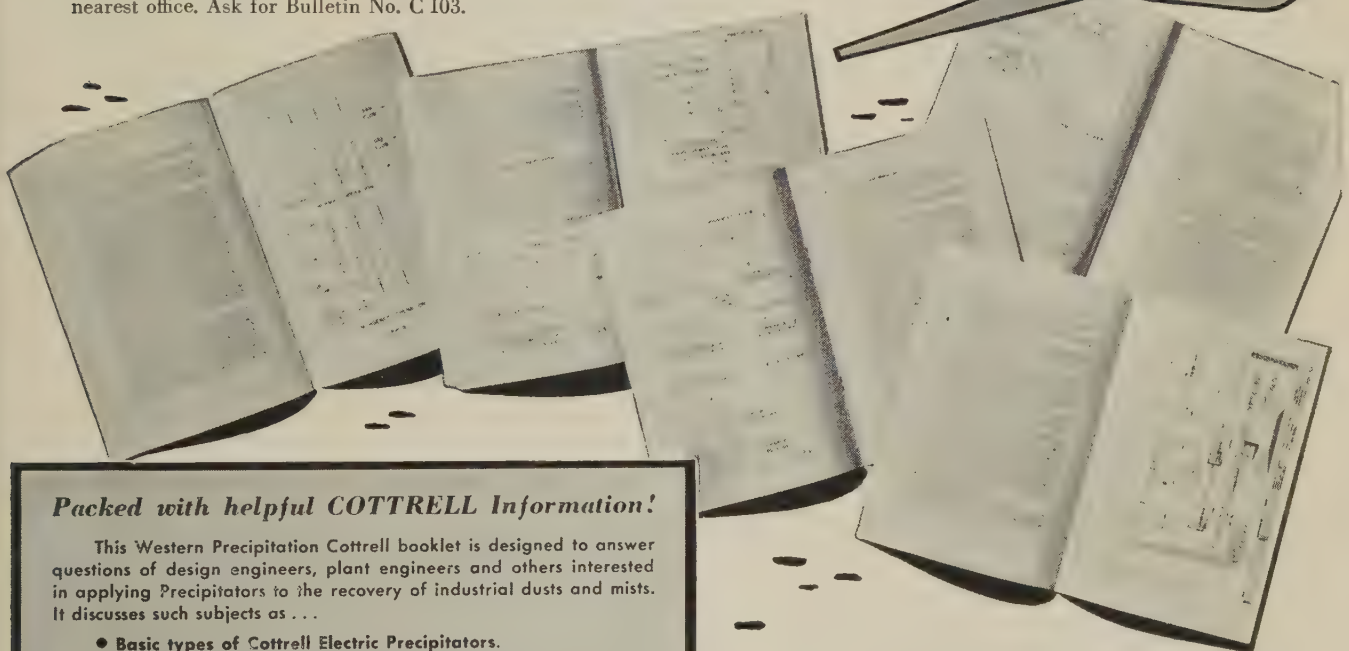

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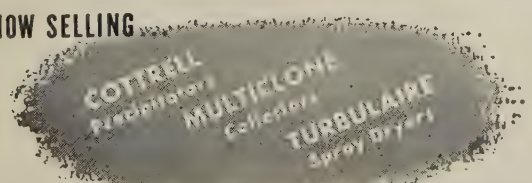

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Capacitors for Industry. By W. C. Bloomquist, C. R. Craig, and R. M. Partington, General Electric Co., and R. C. Wilson, Trumbull Electric Mfg. Co. John Wiley & Sons, Inc., New York, 1951. Cloth, 6 × 9, 246 pages. $4.50.

This book is one of a series written by General Electric Co. authors for the advancement of engineering practice. It presents the practical application data used daily in industry. Among the subjects treated are power-factor fundamentals, location of capacitors, their application to motors and generators, automatic control, operating characteristics, and installation and maintenance.

Viscosity and Plasticity. By E. N. da C. Andrade, Professor of Physics, University of London. Chemical Publishing Co., New York, 1951. Cloth, 5 1/2 × 8 1/2, 84 pages. Illustrated. $2.25.

This book consists of three lectures by the author on viscosity and plasticity given to postgraduates under the auspices of the London Section of the Oil and Color Chemists Association. The chapters deal with the nature and theories of liquid viscosity; the flow of simple liquids, suspensions and gels; and the flow of solids. A good grounding in these subjects would be helpful in understanding this fairly advanced presentation.

Patents for Technical Personnel. By Worth Wade, American Viscose Co. Chemonomics, Inc., New York, 1951. Paper-bound, 6 × 9, 40 pages. $3.00.

This brief monograph has been prepared by the author to give supervision and technical employees information on the function of the patent department. Without doubt the booklet could be expanded to the size of an imposing tome. Instead it looks like the notebook that a reader would prepare after reading a large text and extracting the information that is really needed. It could be written so concisely and surprisingly completely only by an author who has himself read tomes and had a world of experience in the field of patents.

Maintenance Hints. Westinghouse Electric Corp., East Pittsburgh, Pa., 1951. Fabricoid, 3 1/8 × 6 3/4, loose-leaf. 26 sections. $2.00.

This loose-leaf device consists of a series of maintenance hints issued by Westinghouse to assist customers in the maintenance of their electrical production machinery to insure longer trouble-free life. It covers electrical units such as motors, insulation, bearings and lubrication, transformers, circuit-breakers. It should be a must in the library of any electrical engineer or chief electrician.

Paint Film Defects. By Manfred Hess. Reinhold Publishing Co., New York, 1951. Cloth, 5 1/2 × 8 1/2, 561 pages. Illustrated. $12.

This is the first English edition of the standard German work on the subject of the causes and cures of paint fiber defects. It has been prepared for paint users and manufacturers. It is essentially a handbook. Its 88-page index indicates the vast number of ways that a paint work or lacquering can be spoiled despite the fact that great care and skill and the best materials may have been used. A key to the use of the text is given by the author. Faults developed during storage, after application, and when in use are the major divisions but there are 124 subdivisions of importance. It is an excellent handbook.

Process Heat Transfer. By Donald Q. Kern, Patterson Foundry & Machine Co. McGraw-Hill Book Co., New York, 1950. Cloth, 6 × 9 1/4, 871 pages. Illustrated. $8.00.

This is a textbook giving systematic instruction in the calculations, derivation, and empirics of heat transfer, using the language and methods of industry. It is a relatively complete manual for solving the problems of thermal design arising in the process industry such as pulp and paper manufacture. It deals with such subjects as conduction, connection, radiation, heat exchanges, condensation of vapors, cooling towers, furnace calculations, and control of temperature and related process variables.

Encyclopedia of Chemical Reactions. Vol. IV. By C. A. Jacobson. Reinhold Publishing Co., New York, 1951. Cloth, 6 × 9, 790 pages. $14.

The fourth volume of the Encyclopedia continues a discussion of the chemical reactions alphabetically: Iron, Lead, Lithium, Magnesium, Manganese, Mercury, Molybdenum, and Neodymium. The information furnished should save the reader much time and assure accuracy in considering almost any reaction involving these elements. The editor and his associates are making a tremendous contribution to all chemists.

The Pulp and Paper Industry in the U.S.A. Columbia University Press, New York, 1951. Cloth, 6 1/4 × 9 1/2, 378 pages. $6.00.

This book was prepared by a mission of European pulp and paper experts who visited the United States in 1950 under the auspices of the Organization of European Economic Cooperation and the E.C.A. Its members were from Austria, Belgium, Denmark, France, Germany, Greece, Iceland, Ireland, Italy, Luxemburg, The Netherlands, Norway, Portugal, Sweden, Switzerland, Trieste, Turkey, and the United Kingdom.

Its subject matter covers statistics, raw materials, financial situation, technical research, manufacture of pulp and paper, converting operations, labor relations, machinery, etc., as viewed by this foreign group of papermakers.

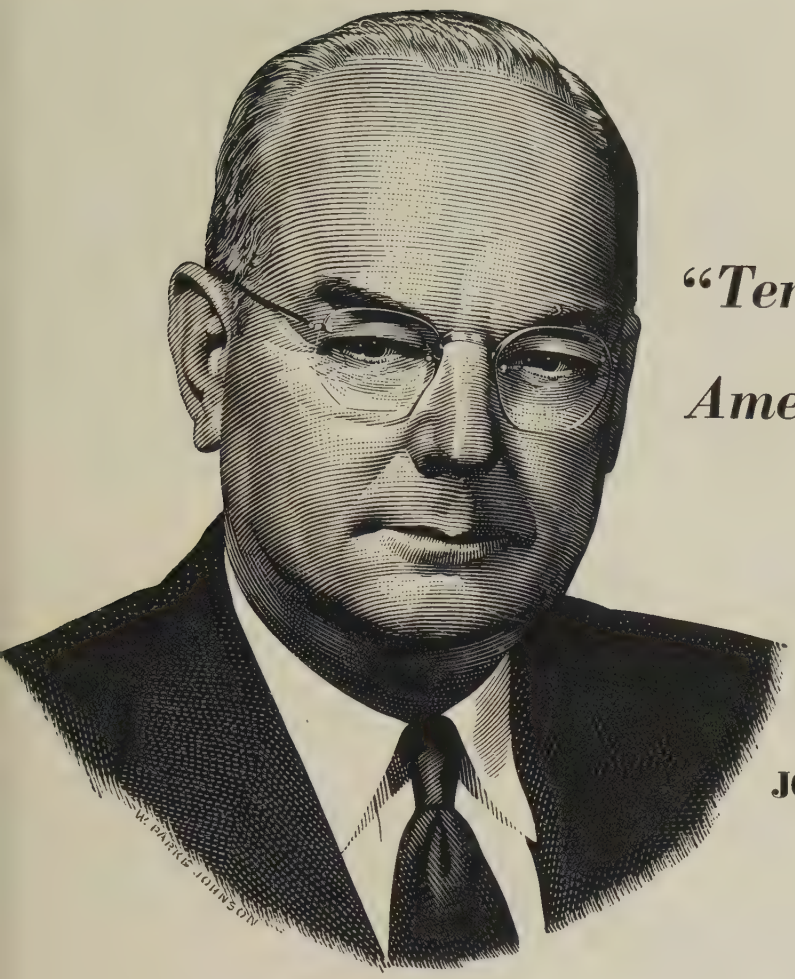
Mineral Wool Insulation Specifications and Standards. Compiled by the Industrial Mineral Wool Institute, New York, 1951. Fabrikoid, 6 1/2 × 9 1/2. $3.20.

This is a loose-leaf device containing 21 specifications relating to mineral wood. The major sections are: Federal Specs.; Navy Specs.; Maritime Specs.; A.S.T.M. Specs.; and Commercial Standards. The subjects covered are: Types of Products, Characteristics, Construction, Selection, Standard Dimensions, Temperature Limits, Thicknesses, Auxiliary Materials, Application Techniques, and Testing Methods.

Statistical Engineering in the Chemical Process Industries. By James R. Thomsen. Chemonomics, Inc., New York, 1951. Paper-bound, 5 1/2 × 8 1/2, 48 pages. $2.00.

This pamphlet is Series 1 on Statistics and is a compilation of abstracts of selected articles dealing with quality control. There are several items relating to pulp and paper. It is essentially a guide to the literature.

Note: Books reviewed in this section may be obtained from the Book Department of the Technical Association of the Pulp and Paper Industry, 122 East 42nd Street, New York 17, N. Y.



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TAPPI DIVISIONS AND COMMITTEES

Recent Reports of Activities

Electrical Engineering Committee

The following is a report of the activities of the Electrical Engineering Committee:

Savannah Meeting. In the general session on Tuesday, Oct. 16, 1951, Walter Mikelson presented his report on the Power Requirements for Fourdrinier Sections of Paper Machines which is part of an authorized committee project for all sections of the machine, and E. W. Davis presented a paper on Properties of Synthetic Rubber and Plastics as Insulation and Sheets on Electric Cable which is part of an authorized project on cable under the chairmanship of A. O. Mortenson.

Mr. Mortenson was to have presented a preliminary report for his Cable Subcommittee which contained a large amount of valuable data, but unfortunately time did not permit this and he graciously gave his place to his guest, E. W. Davis. Mr. Mortenson's report will be published in *Tappi*.

During the round-table session later in the day, R. R. Baker presented his preliminary report on the Power Requirements of Press Sections which is part of the general project on paper machine power requirements. Mr. Baker's final report will be given at the Chicago meeting next Fall.

Discussion. There was not a great deal of discussion either on Mr. Davis' paper or on Mr. Baker's report. There was considerable discussion on Mr. Mikelson's report which is available in detail to those interested since substantially all of it was recorded. In substance, the feeling was expressed by some members of the round-table group that it would be desirable if the flat box vacuum did not have to be known to establish the power requirements of fourdriniers. It was hoped that some way could be found instead to use the kind of paper being made, its freeness, and the water content of the sheet leaving the wire. Mr. Mikelson stated that this might be better, but he knew of no way to accomplish it. He suggested that any of the people present submit ideas for doing so with supporting data. He further stated that a large quantity of data would be required for this indirect approach since it would be empirical; on the other hand, there is more and more interest in the variables which affect water removal and flat box instrumentation is improving so that more data with greater reliability should be available in the future.

There also was some opinion that Mr. Mikelson's data on which the report is based (11 different fourdriniers) were not conclusive. Mr. Mikelson said that he wanted, of course, all the additional data anyone would give him, but he felt that the power formula developed in the report was based on a rational analysis, rather than empirical, and since the resultant data checked the experimental data fairly well, a great deal of experimental data would not be needed.

Committee Organization

The membership of the Electrical Engineering Committee is as follows:

R. R. Baker, Westinghouse Electric Corp., East Pittsburgh, Pa.
E. L. Cowan, Gaylord Container Corp., Bogalusa, La.
R. W. Davis, Allis-Chalmers Manufacturing Co., Norwood, Ohio
T. Foulkes, E. B. Eddy Co., Ltd. Hull, P. Q., Canada
A. S. Goodrich, Hammermill Paper Co., Erie, Pa.
Ray Foster, Champion Paper & Fibre Co., Canton, N. C.
G. W. Knapp, General Electric Co., Schenectady, N. Y.
A. O. Mortenson, St. Regis Paper Co., Box 62, Cantonment, Fla.
M. J. Osborne, Crossett Paper Mills, Crossett, Ark.
Henry J. Perry, Lockwood Trade Journal Company, Inc., 15 W. 47th St., New York 19, N. Y.
J. L. Van Nort, Reliance Electric & Engineering Co., Cleveland, Ohio
R. F. Sorenson, Union Bag & Paper Corp., Box 570, Savannah, Ga.

Proposed Program for 1952 Chicago Meeting

General Session

1. Project Report—Selection of Cable for Pulp and Paper Mill Applications, by A. O. Mortenson, Chairman of Subcommittee.
2. Project Report—Power Requirements for Press Sections of Paper Machines, by R. R. Baker, Chairman of Subcommittee.
3. Paper (Tentative)—Experience with Grounded Power Systems. (Author to be designated later.)

Round-Table Discussion

1. Preliminary Project Report—Power Requirements for Chippers, by M. J. Osborne, Chairman of Subcommittee.
2. Preliminary Project Report—Power Requirements for Supercalenders, by J. L. Van Nort, Chairman of Subcommittee.

Authorized Committee Projects

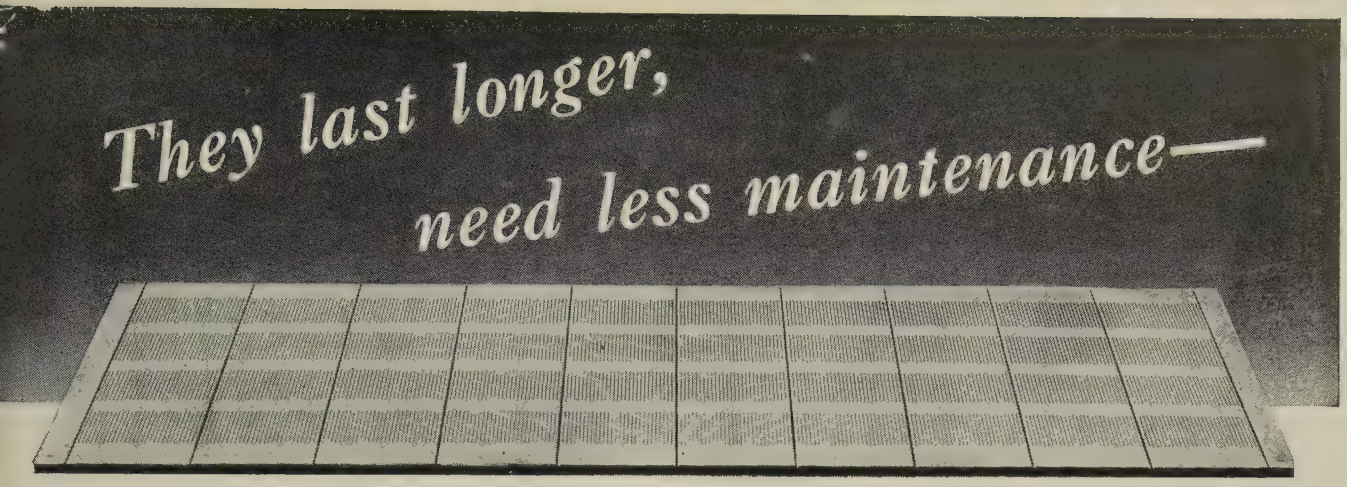
In addition to the authorized committee projects shown in the 1951 *Tappi Year Book* (page 31), the following two projects should be added: Power Requirements for Chippers and, The Selection of A.-C. Motor Control.

We can report progress on our committee projects as follows:

Power Requirements for Paper Machines (No. 358)

Mr. Mikelson has now given his final report on Power Requirements for fourdrinier sections, which of course is still subject to review and amendment at a later date as

Electrical Engineering Committee



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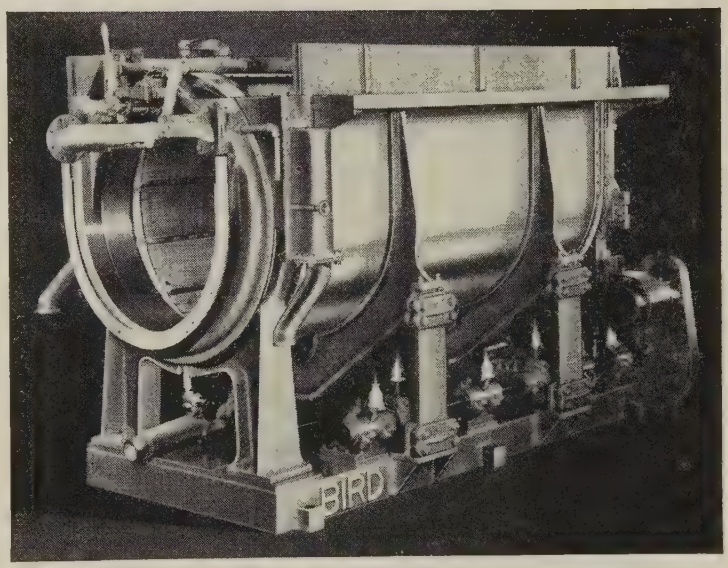

Inconel screen plates in position on a Bird machine. Photo courtesy Bird Machine Co., South Walpole, Mass.

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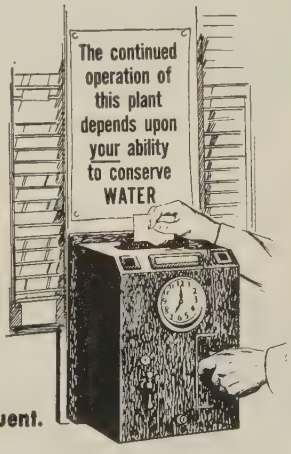

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additional data are disclosed. Mr. Baker has given his preliminary report on the Power Requirements for Press Sections, and will give his final report in Chicago next Fall. In the future, sometime after the Chicago meeting, Mr. Mikelson has been asked to report on the Power Requirements for Paper Machine Driers and Mr. Baker on Paper Machine Calenders. These men have been acting as subcommittee chairmen for their reports and have been aided substantially by many people in the industry working with them, but unfortunately space does not permit listing them all.

Application and Design Characteristics of Motors (No. 359)

This investigation has been headed up by a subcommittee under R. F. Sorenson and it is expected that a final report will be available for publication early in 1952. An excellent job has been done in the phase of the report dealing with kraft mills by Messrs. Sorenson and Foster, and W. V. Knight of Gaylord Container Corp. Mr. Sorenson is now in the process of working up the sections on sulphite and groundwood mills.

Power Requirements for Supercalenders (No. 368)

Because of the pressure of other committee work, nothing has been done on this project as yet. However, J. L. Van Nort has now been appointed Subcommittee Chairman and expects to have a preliminary report for the Chicago meeting in October.

Application of Electric Cable (No. 369)

The Subcommittee on Cable under the direction of A. O. Mortenson has been doing an outstanding job. Substantial progress has already been made by it as reported in the September, 1951, issue of *Tappi*, pages following 75 A. It is expected that an interim report, which Mr. Mortenson was to have given at Savannah as described above, will be published in *Tappi* soon and a final report given during the general meeting in February or at the next Engineering Conference in October, 1952.

Power Requirements for Chippers

This is a new project which has no identifying number as yet. The subcommittee making the investigation will be headed by M. J. Osborne and it is planned to have a preliminary report at the Chicago meeting next October.

The Selection of A.C. Motor Chippers

This is a new project which has no identifying number as yet. The Subcommittee making the investigation will be headed by R. W. Foster and it is planned to have a preliminary report at the Chicago meeting next October.

G. W. Knapp, *Secretary*
Electrical Engineering Committee

Engineering Research and Machine Design Committee

The Engineering Research and Machine Design Committee met at the General Oglethorpe Hotel on Monday, Oct. 15, 1951.

The regular session of the convention on Monday morning was turned over to the Committee. John Lyall presided as Chairman of the meeting. One paper was presented on Capacity of Thickeners, by Robert Nivison of the Improved Paper Machinery Co. of Nashua, N. H.

The regular luncheon of the Committee was held in the Wilmington Room of the General Oglethorpe on Monday at 12:30 with 24 members and guests in attendance. After lunch the present and future programs of the Committee were discussed under the chairmanship of John Lyall.

The committees on Centralized Control of Stock Systems, Correlation of Various Types of Plastometers, and Methods

Engineering Research and Machine Design Committee

for Quickly Estimating Costs of Engineering Projects were scheduled for the afternoon discussion and these topics were discussed only briefly. George Clink, Chairman of the Subcommittee studying Standardization in the Paper Industry did not have work to report on at this time. It was suggested that a study be made of the felt tensions now in use on machines to set up a standard for felt tension. This would necessitate the need for an instrument or device for measuring felt tension and perhaps lead to a study of the effect of felt tension on the life of felts. It was agreed that this matter would be investigated.

Mr. Lyall asked for a vote on a number of possible projects for future work by the Committee. One subject, Constant Tension Devices, was selected for investigation. This project will be explored by George Ehemann assisted by Robert C. Berger and Russell Irwin and a report will be made at the annual TAPPI convention in February.

On Monday afternoon at 3:00 p.m. an open discussion was held by three of the subcommittees in the main meeting room of the hotel.

On Centralized Control of Stock Systems Robert Berger presented slides on various methods used for remote control including those of other industries. After the slides, the Chairman of the Subcommittee, Lawrence A. Moore, discussed the ramifications of the project and asked for comments or criticisms from the audience. The program for this Subcommittee for next year will be a further exploration of this subject with descriptions of existing systems and the benefits derived from their use.

The Subcommittee on Correlation of Various Types of Plastometers under the chairmanship of Kenneth Brenner had a paper presented by Mr. Leighton of the Stowe-Woodward Co. on a study of the various types of plastometers. Also, the new Pusey & Jones plastometer was exhibited by Mr. Hodges of Pusey & Jones. The future program for this Subcommittee will include an investigation of methods for measuring press roll nips and also the effect of width of nip on the quality of the sheet.

Harold Ingraham, the Chairman of the Subcommittee on Methods for Quickly Estimating Costs of Engineering Projects, and Lloyd Ewing discussed the pitfalls of using past costs for future projects but indicated that it can be done if properly approached. They asked for comments and criticisms from the audience on their project. The program for next year will be a method for estimating conveyor or pipe costs.

The future program above and papers for the next Fall meeting will be discussed at the Annual Meeting in February.

R. T. De Pan, *Secretary*
Engineering Research & Machine Design Committee

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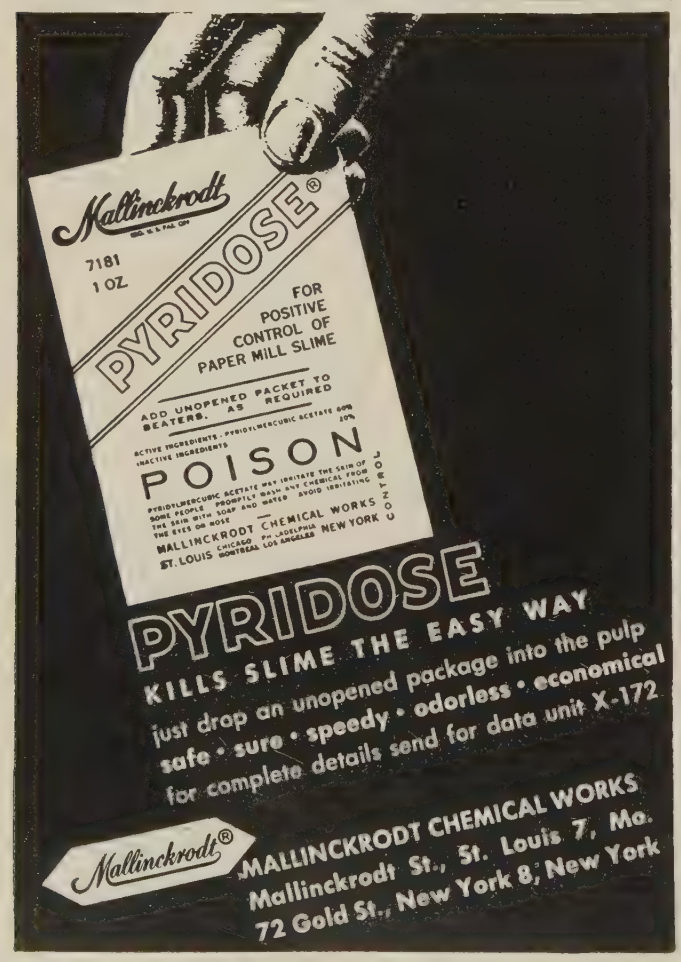
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Materials Handling Committee

The meeting of the Materials Handling Committee was called for 12:30 p.m. in the Wilmington Room of the General Oglethorpe Hotel on Tuesday, Oct. 16, 1951. In the absence of the Chairman, A. P. Schnyder of Ebasco Services, Inc., the Secretary, W. R. McNally of Link-Belt Co., acted as Chairman *pro tem*, and Dan Patch of the Morton C. Tuttle Co. was called on to act as Secretary *pro tem*.

In addition to the above, the following men were present:

G. Cannie, Continental Paper Co., Ridgefield Park, N. J.
C. E. Green, Link-Belt Co., Atlanta, Ga.
D. D. Hall, E. B. Eddy Co., Ottawa, Canada
F. D. Helversen, Crown Zellerbach, Portland, Ore.
J. W. Hemphill, Johns-Manville, New York, N. Y.
V. C. Jones, Continental Paper Co., Ridgefield Park, N. J.
J. M. MacBrayne, Union Bag & Paper Corp., Savannah, Ga.
W. A. McKenzie, Simpson Logging Co., Shelton, Wash.
B. G. Morgan, Hammermill Paper Co., Erie, Pa.
K. C. Neuenfeldt, Hammermill Paper Co., Erie, Pa.
H. F. Parker, New York & Pa. Co., Lockhaven, Pa.
W. H. Rambo, Consulting Engineer, Portland, Ore.
H. A. Stoess, Jr., Fuller Co., Catasauqua, Pa.
M. Strelitz, Continental Paper Co., Ridgefield Park, N. J.
Curt Weil, Ebasco Services, Inc., New York, N. Y.

Mr. Hemphill made an appeal for more active members in committee work.

Mr. Helversen suggested that the committee put some study into the subject of Unit Loads.

Mr. McNally called attention to the fact that to date much of the attention of the Committee had been put on the handling of pulpwood but that there were many other materials to be handled.

Mr. Helversen suggested that it was possible that the Committee could arrange for an interesting paper on Materials Handling by contacting the man who handles this activity for the "Safeway" stores.

Don Hall of Ottawa described the work of the Canadian Committee and their discussion as to the scope of their field. They had arrived at the conclusion that they should have purview of the handling of raw materials from the time they arrived at the mill on a common carrier through processing and as finished goods until delivered to common carrier leaving the mill. They discovered some overlapping with the work of other committees. For instance, pulpwood handling overlapped some of the Forestry or Woods Committee activities. Pulp handling overlapped the Sulphite Committee activities.

Someone suggested that the TAPPI Materials Handling Committee consider its field to be from Gate to Gate. This would rule out the study of bundling and loading of pulpwood in the woods, which has a big bearing on its handling in the yard.

The Canadian Committee have published several reports and have found that they have to be careful about naming particular makes of equipment in such publications. They have a 12-man committee.

Mr. McNally asked for suggestions for subjects to be discussed at the round-table discussion to be held on Wednesday afternoon, under the possible subdivision of wood, chips, chemicals, and finished paper.

Mr. MacBrayne stated that the materials handling studies at the Union Bag & Paper Corp. had started in their bag department and were working back through the paper and pulp departments toward the wood end of the flow line. He stated that a moving picture of some of their practices would be shown during the barbecue at the Golf Club on Thursday.

Curt Weil suggested that questionnaires be sent to the various mills to determine what problems were uppermost. Mr. McNally cited the difficulty the Committee had faced in getting replies to questionnaires. Mr. Cannie suggested that one important materials handling field was waste disposal.

Mr. McKenzie felt that the Committee should formulate a

policy as to what our field embraced. He felt that questionnaires should be so made up that they could receive "Yes" or "No" answers.

Mr. McNally stated that he believed the aim of the Committee was to establish standards in the industry.

Materials Handling Committee

Mr. Parker felt that any questionnaires should be specific and should be followed up. He believed that pumping, which is really a materials handling problem, should be omitted from our program as it would be handled by the Hydraulics Committee. He thought the Committee should have a definite program..

The Secretary *pro tem* suggested that those present did not represent many mills as many were from supplier or service companies but that it might help the Committee catalog the problems if each mill representative present would send to Mr. McNally a complete list of the materials which presented handling problems in his mill.

Mr. Neuenfeldt suggested that case studies were valuable and that a paper might be presented describing some special case of materials handling which was particularly successful.

Mr. McKenzie stated that the value of materials handling experiences was not of much value unless the economics involved were included.

Mr. Helversen again referred to Unit Loading, calling attention to the fact that it is related to sales and especially to the Army and Navy market.

Both Mr. Helversen and Mr. McKenzie complained that the Committee had not accomplished anything. Mr. Parker stated that the Mill Maintenance Committee felt the same way about their efforts in Committee but he felt that some real good had been accomplished by their papers.

Mr. McKenzie moved that Mr. Helversen write up the method of handling wet pulp at West Linn, with illustrations.

Mr. McKenzie suggested a definite questionnaire on the minimum height of drop for a definite length of pulpwood at a transfer point.

The round-table meeting of the Materials Handling Committee was conducted with W. R. McNally acting for A. P. Schnyder as moderator.

The following persons were in attendance:

W. R. McNally—Link-Belt Co., Chicago, Ill.
F. M. Burt—Raybestos-Manhattan, Inc., Birmingham, Ala.
F. S. McCall—Union Bag & Paper Corp., Savannah, Ga.
C. E. Wills—Halifax Paper Co., Roanoke Rapids, N. C.
F. D. Helversen—Crown Zellerbach, Portland, Ore.
H. A. Stoess, Jr.—Fuller Co., Catasauqua, Pa.
D. D. Hall—E. B. Eddy Co., Ottawa, Canada
W. A. McKenzie—Simpson Logging Co., Shelton, Wash.
H. F. McLeroy—Union Bag & Paper Corp., Savannah, Ga.

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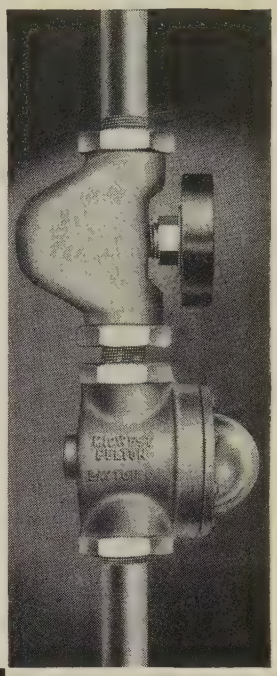

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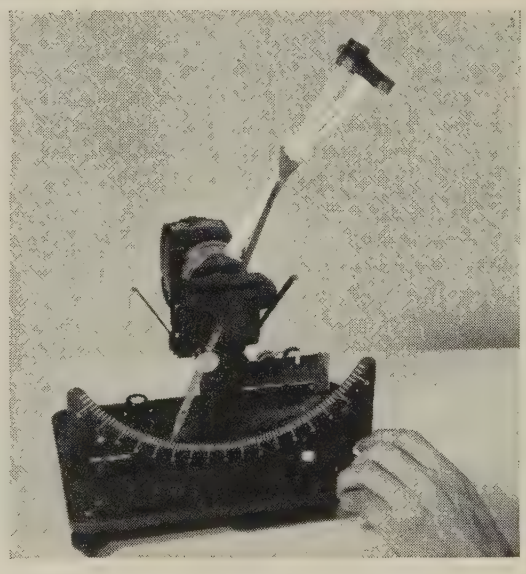

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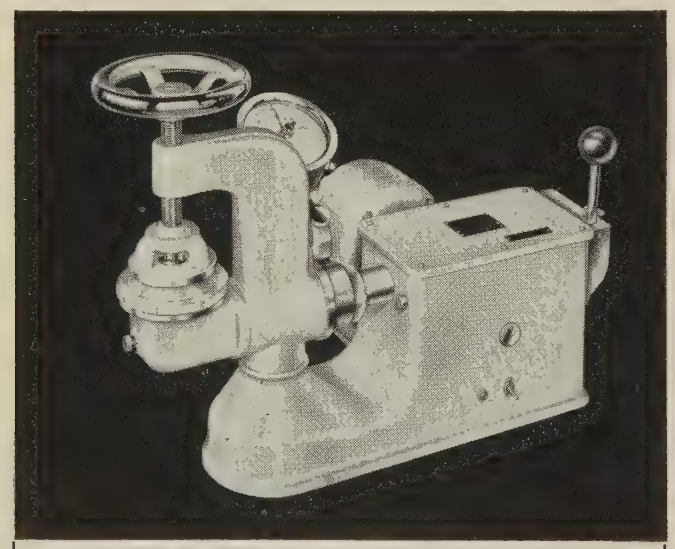

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F. W. Taylor—Hewitt-Robbins, Jacksonville, Fla.
E. J. Fadden—Mead Corp., Chillicothe, Ohio
W. T. Bennett—P. H. Gladfelter Co., Spring Grove, La.
W. H. Dolley—International Harvester, Jacksonville, Fla.
C. E. Patch—Morton C. Tuttle Co., Boston, Mass.
E. H. Barclay—Union Bag & Paper Corp., Savannah, Ga.
Alex C. Ormond—Union Bag & Paper Corp., Savannah, Ga.

The discussion period was divided into two parts, one lasting for about 30 minutes consisted of questions by the audience referring to papers presented during the morning session. The authors of these papers gave appropriate answers.

The balance of the program was concerned with the difficulties involved in handling pulpwood from the woodlot to the wood yard. In this phase, discussion was divided as follows 1—Rail; 2—Truck; and 3—Boat.

Rail. It was decided that eventually, materials becoming available, it would pay the paper mills to band together and to work with the railroads to do away with pulpwood shipments in box cars and to make all shipments by rack cars, since there is no economical way to unload box cars. It was pointed out that from studies conducted in a number of paper mills that the man-hour productivity rate, brought about by manually unloading these cars, reached an astonishing low.

The possibility of designing a small portable conveyor for use in these cars was decided to be an unsatisfactory project.

The use of specially designed cars, for use with special car dumpers for unloading pulpwood, was not favorably received since it becomes too expensive a proposition for the common benefit of many mills.

In addition to the unloading economics involved, it was pointed out that better methods of handling wood at the source, as well as at the loading depot, would be reflected in additional economies at the woodyard unloading areas.

Trucks. Although the hauling radius varies with the mill, much of the so-called local pulpwood arrives at the mill by truck. The problems involved in the use of trucks revolve about the lack of standard loading methods and, therefore, the difficulties involved in designing and operating suitable truck unloading devices.

It was decided that it would be advisable to foster a program of standardization on truck loading, which, in turn, could lead to more satisfactory unloading procedures—both for speed and economy.

A representative of a leading truck manufacturer volunteered to furnish data they had gathered on this project themselves, as well as designs of trucks to improve this condition, for use of the Committee on this investigation.

Boat. Since this problem is not common to many mills, it was decided that those mills faced with this particular problem would work with the Committee directly.

The whole procedure of this round-table discussion was to bring out into the open facts and questions pertinent to the case study of the Materials Handling Committee, subcommittee headed up by W. A. McKenzie on the Economics of Wood Handling from the Woodlot to the Woodyard.

C. E. Patch, *Secretary, pro tem*
Materials Handling Committee

Steam and Power Committee

The Steam and Power Committee met at Savannah, Ga., on Oct. 16, 1951. The main purposes of this meeting were: to select new officers and to select the program for next year.

The new officers are: H. R. "Russ" Arnold, *Chairman*, and J. "Jack" Gottlieb, *Secretary*.

The new members of the Committee are: George W. Bollinger, West Virginia Pulp & Paper Co.; Edwin Leiss, Continental Paper Co.; and W. B. Wilson, General Electric Co.

Next year's meeting will be in Chicago the second week of October. Here are the subjects selected:

Main Program

1. Electrical Versus Mechanical Drives for Paper Machines. No doubt this broad subject will have to be combined with the Electrical Committee and may be the whole session.

Steam and Power Committee

2. Application of Package Steam Generating Units to the Paper Industry.

Round-Table Meeting

1. Solid Fuel Handling Equipment.
2. Relative Advantages of Pulverized Coal and Stoker Firing.

These two items are to be considered as a single over-all topic. Although both of these were suggested for a main meeting the Committee felt these were better for a round-table discussion.

Long-Range Topics

We ran short of time and did not get to this subject. However, based on the returns of the questionnaire the replies were overwhelming in favor of collection of steam and power data. This topic which I am phrasing for content is Collection of Industry Data of Steam and Power Requirements per Unit of Product in the Over-all and Various Steps of Manufacture.

I want personally to thank all of the members of the Steam and Power Committee for the fine and prompt cooperation these past few years.

W. C. Bloomquist, *Secretary*
Steam and Power Committee

Chemical Engineering Committee

The Chemical Engineering Committee met at the General Oglethorpe Hotel, Savannah, Ga., on Oct. 15, 1951. The members present were: J. R. Lientz, *Chairman*, J. J. Goss, L. C. Jenness, and H. O. Teeple. The members of the Digester Corrosion Subcommittee present were: J. R. Lientz, *Chairman*, S. J. Baisch, F. W. Flynn, J. J. Goss, W. J. Shelton, W. Smith, and H. O. Teeple.

This Luncheon Meeting was called to order by J. R. Lientz at 12:30 p.m. Two items of business were discussed.

The first had to do with the program of this Committee during the forth coming February TAPPI meeting. It had been decided previously that the Chemical Engineering

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Committee and the Alkaline Pulping Committee would jointly sponsor a Session on Semichemical Pulping and certain members of the Committee agreed to work up papers on the several phases of the general subject.

The second item, that of the Digester Corrosion Subcommittee, was discussed insofar as its current status was concerned. It was pointed out that a Subcommittee meeting was held in Chicago on September 23 and 24 and out of that meeting came the conclusion that additional help is desirable in analyzing the Questionnaire Data if a good job is to be done. Further, several steps need be taken to keep the digester information up to date. Some thoughts had already been formulated on these two points which were brought up for discussion at this Committee Meeting. As a result, it was decided to approach TAPPI for limited financial assistance in having an appropriate outside organization evaluate the questionnaire data obtained to date.

Chemical Engineering Committee

In connection with keeping the digester information current, the matter of the issuance of a follow-up questionnaire was discussed. It was felt this could be postponed for awhile until some conclusions could be drawn from the evaluation of the questionnaire data, particularly with respect to gaps in the data.

In an effort to provide the alkaline pulping mills with a suitable procedure for recording information on the corrosion of digesters, the Subcommittee is currently preparing a Data Sheet on this subject.

The meeting was adjourned at 2:00 p.m., by J. R. Lientz.

H. O. Teeple, *Acting Secretary*
Chemical Engineering Committee

Digester Corrosion Subcommittee

Following is the Second Progress Report of the Activities of the Digester Corrosion Subcommittee presented on Oct. 15, 1951, at Savannah, Ga.

The Subcommittee has made further progress since the issuance of the First Progress Report, Feb. 21, 1951, and this Report will be a continuation in that further questionnaire data will be presented.

The Digester Corrosion Questionnaire was sent out to those mills not covered in the first issue. Thirty-eight were sent out and of these nineteen were returned. The picture to date of all questionnaires is shown in Table I.

It is to be observed that the coverage obtained is good. However, the number of digesters for which corrosion rates were reported, 143 out of 345 or 42%, indicated a lack of specific corrosion data on the part of a considerable number of mills.

It is the belief of the Committee that a greater effort on the part of the mills to obtain performance data on their respective digesters would be of considerable assistance in scheduling digester replacement and also would indicate to the mill in a real sense the seriousness of the digester corrosion problem. To assist the mills in this respect, the Committee has arranged to provide a sample form wherein digester performance data can be conveniently shown. This form was developed by a cooperating mill and is the standard form now for that mill, attached hereto.

Table I. U. S. Mills Replying to Digester Questionnaires

	Kraft	*Soda*
South	23	0
Northeast	0	6
North Central	7	2
Northwest	7	1
Total	37	9
Questionnaires sent out	50	20
U. S. mills	59	20
Returns—mills	37/50 or 74%	
Kraft coverage	37/59 or 63%	
Soda coverage	9/20 or 45%	
Alkaline coverage	46/79 or 58%	

This report will follow the same outline observed in the First Progress Report in that the report will consist of several sections.

A. Kraft Stationary Digesters

The total number of digesters reporting are 299 which include replaced and existing digesters. Of these, corrosion rates were reported for 137 digesters. While some high rates are shown, it is interesting to note 73% of the reported corrosion rates are less than 50 mils per year, less than $^1/_{16}$ inch.

The majority of the digesters which were reported were made of either A-70 or A-285 steel. There were a number made of A-212 steel, and a few of A-89, a low carbon-low tensile steel. About one third of the reported digesters showed no steel specification. These were mostly old digesters which had been replaced.

Sulphidities appeared to range between 20–25% in the main. There were some reported trends to the lower sulphidities.

B. Kraft Rotary Digesters

There were eight additional rotary digesters reported, making a total of 65. Of these 8, 3 riveted failed in 4 years, 3 hammer welded lasted 11 years, and 2 A-70 fusion welded are in service after 13 years. Sulphidity was 31% (active basis). In general, the rotary digester picture does not appear to be too different from that of the stationary digesters.

C. Soda Digesters

An additional 19 digesters were reported, making a total of 51. The fact that corrosion does not appear to be a problem in the case of soda digesters was further exemplified by the reported information. For example, one digester installed in 1898 and three in 1899 are still in service.

D. Canadian Kraft

Because of excellent cooperation with the Digester Corrosion Group of the PPRIC, the TAPPI questionnaires were sent to a number of Canadian mills and were turned over to the Committee for examination. These questionnaires reported 60 digesters with corrosion rates ranging from 0.013 Ipy to 0.070 Ipy, a range not too different from the majority of U. S. kraft digesters.

Steels used in the digesters were reported A-201, A-70, A–89, and A–285.

Sulphidities appeared to run higher than those reported for U. S. kraft mills but there is the matter of the basis used for the calculated sulphidity which must be considered before accurate comparisons may be made.

E. General Comments

One important comment is to correct a statement made in the First Progress Report as to the rate of corrosion currently in effect for a type 410 clad steel digester. A recent Audigage measurement indicated a rate of corrosion of about 2 or 3 mils per year as compared with 84 mils per year reported previously. The correction is based on the second Audigage reading and also upon an error made in calculating the initially reported rate.

A recent inspection of a type 316 Cb clad digester, after 12 months' service, indicated some corrosion appeared to be occurring in the vapor space above the liquor level. This corrosion was reported as slight pitting.

A recent inspection of an Inconel clad steel digester indicated excellent performance after about one year of service.

Stainless steel linings in digesters in two mills reported repairs necessitated by cracking of the lining.

In furtherance to the subject of digester linings particularly alloy, a number have been installed all over the United States. Some of these linings have been shop applied and some have been field applied. Some of the shop-applied linings have been successful and some have not. In the latter cases, field repair linings have been attempted, some of which were successful and some were not. The amount of real knowledge of field lining procedures has increased during the past couple of years which should be helpful in the future. Sources of information on lining procedures should be carefully sought because specific circumstances are a large factor in the eventual success of the lining.

Other linings, such as carbon brick, have been installed in some digesters. One mill reported plans to carbon brick line six digesters. A recent inspection of a carbon brick lined digester indicated the lining in general was in excellent condition after about one year's operation. However, some slight spalling had occurred on several courses near the top of the shell section.

With respect to the comment in the First Progress Report as to the apparent superiority of the A–89 steel over an A–70, the report of a Canadian mill provides more data substantiating this point. Three A–89 steel digesters, 0.14% carbon, showed corrosion rates of 22, 17, and 13 mils per year, giving 260, 260, and 240 months' service, respectively. An A–70 steel digester installed about 9 years later showed a corrosion rate of 44 mils per year, with a service life of 170 months. It is to be noted all four digesters were of the same size and with the same corrosion allowance.

The PPRIC, during the course of their work on the digester corrosion problem in Canadian mills, have developed a new instrument for measuring digester walls. This instrument employs Cobalt 60 isotope and can be used to measure thickness from the inside or from the outside. In the latter case it is not necessary to remove the lagging. Further, it is not necessary that the digester be cooled down for the inspection.

Drying and Ventilating Committee

In the first Progress Report some comments were made relating to the procedures to ascertain the extent of digester corrosion. It is the consensus of the Committee that it is extremely desirable for the mills to set up some sort of an adequate procedure. Based upon the returned questionnaires, many mills had no idea at all what the order of current corrosion rates were encountered in their digesters. The questionnaires indicated that more frequent inspections and/or more adequate inspections could be made.

In closing, the Committee wishes to thank the cooperating mills for their assistance in answering the questionnaire. Due to the difficulty in finding sufficient time to analyze the questionnaires, the reports to date are not as complete as the Committee would like them to be. There is much more information in the questionnaires which, when properly summarized, will be of much interest to cooperating mills. The Committee hopes to make available in the near future a more complete analysis of the reported data.

H. O. Teeple, *Secretary*
Digester Corrosion Committee

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Drying and Ventilating Committee

The Drying and Ventilating Committee had a very enjoyable meeting at Savannah on October 15. About twelve in all attended, including eight of our committee members.

We decided to try for more data in relation to our drying rate curves at the higher pressures, 75 pounds or more in the driers. If there is enough material, we might make a real project of the high-pressure drying.

C. A. Young and the Chairman will contact the machine builders, to see how many machines there are, in what locations, and what types of paper or board are made. This will give us an idea of the spread over the industry. We have already written several letters in regard to this.

Mr. Montgomery will edit the translation of the German paper, to get it in shape for publication.

We have one subject that seems likely for next year's engineering conference, and hope to come up with a second subject in the next few months.

MARSHALL L. BARKER, *Chairman*
Drying & Ventilating Committee

Mill Maintenance and Material Committee

Five subcommittees were appointed by Chairman Hubert F. Parker at the meeting of the Mill Maintenance and Materials Committee on Monday Oct. 15, 1951, at the Sixth Engineering Conference of TAPPI at Savannah, Ga. These committees will deal with Screen Plates, Lubrication, Protective Coatings, Electrical Maintenance, and Maintenance Training Programs in Kraft Mills.

In reviewing the points discussed at the Fifth Engineering Conference at Cincinnati the Chairman stated:

That B. L. Merriss of the Oxford-Miami Paper Co. had consented to accept the chairmanship of Subcommittee on Maintenance Costs with the privilege of appointing his committee.

That Henry W. Downs, Jr., of the Union Screen Plate Co. of Canada had agreed to be the chairman of the subcommittee to develop Project No. 376, Screening Capacities of Different Types and Designs of Screen Plates on Various Types of Stock. Mr. Downs was given the privilege of selecting his committee.

That during the past year he had talked with the representatives of several of the manufacturers of rubber rolls, who had pointed out the tremendous variances that they had encountered in grinding rolls for apparently the same duty but for different customers and the extremely conflicting opinions of roll grinder operators as to how rolls for apparently the same service should be ground. One of the Committee members stated that an effort had been made to set up a grinding schedule for the various mills in the organization he represented so that all rolls for a specific job would be ground alike in all mills, but that unless a constant check was maintained on these schedules, there was a tendency on the part of some supervisors to have rolls ground contrary to schedule. The Committee agreed that there was wide variance in roll grinding procedures, but thought that a roll grinding schedule, if set up would be a valuable guide.

That he had talked with several about The Compilation of a Check List for Mechanical Preventive Maintenance of Pulp and Paper Mills and that these talks indicated that the project should be broken down into many subdivisions and that if such work was to be undertaken, it would be helpful to secure the assistance and cooperation of those concerns that published such information during World War II. This project if carried to completion represents an enormous amount of work and could become the basis for a Maintenance Handbook.

That he knew that at least four of the electrical equipment manufacturers had published information which if condensed and unified might become the basis for the companion project, The Compilation of a Check List for Electrical Preventive Maintenance in Pulp and Paper Mills. A. M. Cooper and J. L. Van Nort agreed to gather this information together and see what could be done to develop the check list.

In response to the Chairman's request Charles J. Brown told the Committee that the work on Project No. 342, Copper and Copper Alloys and Monel and Stainless Steel, was progressing and that it was expected that this project would be ready for the Committee meeting to be held in conjunction with the Seventh Annual Engineering Conference at Chicago in October, 1952.

Mill Maintenance and Materials Committee

Mr. Brown also told the Committee that it was expected that a report would be made on the papers which have been written by the National Association for Corrosion Engineers for the Pulp and Paper Industry printed in *Tappi* during the first quarter of 1952.

R. G. Macdonald advised the Committee that specification E400s-43 on Corrosion Resistant Castings which had been very ably prepared by J. A. Lee had been completed.

A. I. Sippola and C. G. Russell Johnson presented their paper Pulp and Paper Mill Lubricants—Specifications. Mr. Sippola requested the Committee members to read the paper carefully and within the next 30 to 60 days send him their comments, suggestions, and criticisms, so that the paper could be put into final form for publication in *Tappi*.

The Chairman reported that a request had been received from the American Society of Lubrication Engineers for a committee to work with them on the types of equipment used in paper mills, the lubrication problems involved in the design developments of such equipment, and the resulting operating conditions which may affect lubrication. He stated that in view of the work already done he was appointing A. I. Sippola, Chairman, C. G. Russell Johnson, and R. H. Smith of Beloit Iron Works as a committee to work with the American Society of Lubrication Engineers.

The Chairman stated that a request had been made for a Tabulation of Protective Coatings for Use Against Corrosive Conditions in Pulp and Paper Mills and that W. W. Henderson of the Earl Paint Co. had agreed to head a subcommittee of his choosing, to undertake this task.

The Chairman reported that in going through the Committee correspondence he had found reference to two papers that had been prepared by the Beloit Iron Works and in following this lead, had secured the Preventive Maintenance pa-

pers entitled "Rubber Covered Rolls—Storage and Handling" and "Cleaning Suction Roll Shells." These papers will be submitted to *Tappi* for publication and the possibility of being made up into data sheets.

In reply to the question "Do Other Kraft Paper Mills Have Maintenance Training Programs?" it was suggested that a report be made to TAPPI on such Maintenance Training Programs as do exist so that the smaller mills could benefit from the experience gained by the larger mills in training older workers and on the job training. A subcommittee consisting of Harry C. Moore, Chairman, F. D. Helversen, and L. C. Crowder was appointed to gather this information and report at a later meeting.

A discussion of TAPPI Data Sheets brought out: That the sheets should be some standard size (Editor's note: they are: $8^1/_2$ x 11″); that they should be of transparent paper with printing on only one side, so that exact reproductions could be made and given to the interested worker, and when torn or dirty could be disposed of; that the sheets should be prepared for filing in a standard loose leaf binder.

The plans for the meeting at Chicago next October are still in outline form, however, they call for two papers and a round-table discussion. The subjects for the papers are Coverings for Rubber Rolls and a discussion of Safety. The topic for the round-table discussion is Storeroom or Toolroom for Maintenance Tools.

HUBERT F. PARKER, *Chairman*
Mill Maintenance and Materials Committee

Hydraulics Committee

The Hydraulics Committee met at the General Oglethorpe Hotel, Savannah, Ga., Tuesday, Oct. 16, 1951. Those present were:

K. J. Mackenzie, *Chairman*, Eastman Kodak Co.
T. Agronin, Shartle Bros. Machine Co.
Frank R. Forest, Allis-Chalmers Mfg. Co.
V. DeP. Gerbereux, Worthington Pump & Machy. Co.
Lyle C. Jenness, University of Maine
George V. Shaw, Ingersoll-Rand Co.
Victor P. Head, Fischer & Porter Co.
R. E. Durst, University of Maine

It was agreed that the paper, An Analysis of the Data on Stock Flow in Pipes, presented at the morning session, by

Hydraulics Committee

R. E. Durst, A. J. Chase, and L. C. Jenness, represented the conclusion of the second phase of the work on pipe friction for which the Hydraulics Committee was originally set up. This paper represented approximately one year's work at the University of Maine covering an attempt to determine whether or not any mathematical relationships existed between pipe friction, pipe diameter, velocity, and consistency. The conclusion of this study as presented in the paper was that mathematical relationships do exist and formulas were presented to cover this mathematical relationship, together with the percentage variation in the data from these formulas.

The paper, A Shear Criterion for the Hydraulic Behavior of Paper Stock in Pumps, Pipes, Valves, and Flow Meters, by Victor P. Head, covered independent work done by the Hydraulics Research Division of the Fischer & Porter Co. It was agreed by the members present that the instrument developed by Mr. Head for the determination of shear could possibly give a friction factor that could be added to the formulas developed above from the Brecht & Heller data so that calculated pipe-friction loss could be determined once the shear characteristics of the particular fiber have been obtained.

It was further agreed that a refined model of Mr. Head's equipment would be built, along with a 1-inch diameter pipe testing setup, to determine whether or not there is any relationship between pipe friction and the shear criterion obtained by Mr. Head's shear tester.

It was further agreed that any test work conducted under the auspices of the Hydraulics Committee should be limited by the following conditions:

Pipe diameters—3 to 12 inches
Stock velocity—2 to 10 feet per second
Consistency—2 to 12% oven-dry

It was further agreed that the nomographs for pipe friction, prepared by Dr. Durst, would be arranged for publication as TAPPI data sheets.

K. J. MACKENZIE, *Chairman*
Hydraulics Committee

Data Sheets Committee

The Data Sheets Committee has not functioned during the past year as it had hoped, either from the collection or production standpoint. Some 25 data sheets are in the national office awaiting processing.

The major portion of these data has been supplied by the Drying and Ventilating Committee and the Electrical Engineering Committee, as the results of engineering projects. Several other committees of the Engineering Division have work in progress which will result in Data Sheets.

During the year Local Section Chairmen were asked to appoint Data Sheet Chairmen for their sections. It was thought that this new setup would arouse more interest within the mills and machinery manufacturers which are the vast reservoirs of valuable information. The time lapse has been too short to judge the effectiveness of this procedure.

The procedure of getting data sheet project approval is easy. An outline of the data to be developed, approximate cost to cover compilation, drafting, etc., the name of the person who will do the work, and the probable number of data sheets should be sent to the Data Sheet Chairman. He will obtain approval from the Committee Chairman and the Division Chairman.

One other major problem besides new data sheets is a review of the old ones. Some are from unknown sources, some are reported incorrect, and an exhaustive review is in order. So far no one has been found with a sufficiently broad background who will undertake this job with or without compensation.

It would seem that with the volume of material that should be available that the Executive Committee should decide if the project is worth while. If so, then a full-time qualified engineer should be put on the job to travel through the industry picking data from the mills and their suppliers, preparing same, and then distributing the information.

HENRY J. PERRY, *Chairman*
Data Sheets Committee

REPORTS AND DISCUSSIONS

Presented at National and Regional Conferences

*Equipment Cost Subcommittee Report**

A PROGRAM to provide a ready means of estimating equipment costs in the paper industry is no small project. To be effective, it should include a study of all or most of the common classes of equipment and should provide a ready means of adjusting the data to economic changes.

Prior to going ahead with a program of such magnitude, it seemed appropriate to select at random one classification of equipment and make a preliminary survey. This would be in the nature of a trial run to determine the availability and reliability of the data; to get an idea of the time required to assemble the information; and to get a concrete expression from the members of TAPPI as to the value to them of this type of information.

The matter of determining the relationship between cost and size of any piece of equipment is complicated by a number of factors. This is particularly true of pulp and papermaking equipment. In the first place, to be of any value, it must be possible to express the size of the unit in terms which are readily available. Frequently, such terms are lacking and size for a given capacity cannot be predicted without more complete data than is generally available.

It is further complicated by the fact that many classes of equipment exist that do the same job but utilize entirely different principles. Usually, in this case, the various types have advantages in the specific job or jobs for which they are intended. To effectively use the type of information we are considering, one must necessarily be familiar with the operating advantages and disadvantages of the various subtypes.

Another consideration is the fact that when a new type of machine is introduced, it usually takes a considerable period of time, particularly, if patent or modifications in design are involved, for the cost stabilizes itself with respect to other equipment.

In order that the results be representative, it was recognized that the type of equipment selected for the trial must be subject to the effects of the above factors. After consideration of a number of items, savealls were selected as being representative in this connection.

For the purpose of this study, the general subject was further subdivided into the following classes: gravity, vacuum, flotation, and sedimentation. Obviously, those types cannot be compared with one another, solely on a first-cost basis, and it is in no way the intention of this report to do so. Each has advantages in the service for which it was designed. Materials of construction vary. Operating costs and efficiencies of the various types are neither uniform nor consistent.

It is felt, however, that the latter elements should not be be considered in this study. First, since both operating cost and efficiency are known to vary widely with conditions, any analysis on the basis of the limited data available might prove to be misleading as well as unfair. The assumption must be made that anyone using this type of information is familiar with the relative advantages and disadvantages of the various types in the application considered. Use of the data should be limited to rough determination of cost of suitable units in the approximate capacity range.

Consideration is given to the fact that such a series of cost evaluations must be made on a common basis. The most useful information would seem to be based on final installed costs. This would include the basic equipment, freight, installation, foundations, wiring, and piping integral to the unit as well as normal insulation, painting, etc. Freight rates, local conditions, and the limitations imposed by the specific project such as space, layout, etc., will frequently have a considerable effect upon the final installed cost. In spite of this, average installed costs are used with the thought that adjustments may be made to the estimated values for any unusual conditions known to exist.

The first classification investigated was vacuum-type savealls. The primary problem encountered was the fact that no simple means of translating capacity to size was readily available. Conditions from one installation to the next vary widely and have such a pronounced effect upon the area required to handle a specific volume of white water, that the expression of size or cost on the basis of volume to be handled was recognized to be impractical. This is true even if the critical variables such as fiber content, freeness, temperature, etc., are known.

Consequently, the only available course was to plot size in terms of area against cost. These data would be useful only to those who through previous experience or special knowledge had a pretty good idea of the size unit required to do the job in question.

The following information was derived from one manufacturer and covers the size range from 8 by 6 to 8 by 16. The cost of the installed 8 by 6 unit in rubber and cast-iron construction, including foundations, freight, installation, integral piping, wiring, and accessory equipment was estimated to be $49,500. As the face width was increased, the cost increased progressively to a figure of $67,000 for the 8 by 16 on the same basis. It is recognized that this gives a very flat curve, with cost variable as the 0.29 power of size, and this is explained by the fact that the increments in size were taken by varying face width at a fixed diameter. This was done as the 8-foot machine is the predominant size in use.

As noted above, the capacity of this type of saveall is very dependent upon furnish and operating conditions. Consequently, it is not possible to express these figures in terms of white water gallonage except in the most general sort of a way. For example, the 6 by 8-foot with roughly 150 square feet might be expected to have a capacity of from 150 to 600 g.p.m, and the 6 by 16 with 400 square feet from 400 to 1600. This range could be narrowed somewhat if the type of furnish is taken into account, and it could be narrowed considerably, of actual drainage tests were conducted. The latter, however, appears to be beyond the scope of this type of analysis.

In the case of sedimentation savealls, the opinion was reached through consultation with one of the leading manufacturers that a fair correlation did exist between size and capacity. This does not mean that all white waters have the same settling characteristics. The usual practice is to adjust the settling properties by the use of chemicals and operating techniques. At the same time, in the case of a poor settling stock, performance standards might be reduced somewhat rather than to go to a larger size unit. Beyond a certain point increases in size are not usually justified by the resultant increases in performance. As pointed out above, the use of this type of information necessitates comprehensive knowledge of the operation of the various types of savealls and their respective suitability in various applications.

* Prepared by Lloyd Ewing, Ewing Engineering Co., Milwaukee, Wis., for the Subcommittee of the Research and Machine Design Committee of TAPPI. Presented Oct. 15, 1951, at the Sixth Engineering Conference in Savannah, Ga.

Since it was felt that sufficient correlation between size and capacity did exist, the following costs are based on white water gallonages. Estimates were made over the range from 350 to 5000 g.p.m. which correspond to diameters of 20 and 75 feet, respectively and a depth of about 16 feet. Roughly, the costs vary as the 0.6 power of the capacity. The range was $21,000 for the 350 gallon unit and $119,000 for the 5000 g.p.m. These costs are for steel construction, and as in the other cases, represent complete installations. With concrete construction these costs would be expected to increase about 5%.

In order to determine whether or not any correlation existed between cost and available indices, 1940 costs were assembled for comparison. During this period an increase of about 20% was noted. Of the various indices available for comparison the Marshall and Stevens Index for Pulp and Paper Manufacture was considered to be the most applicable.

Since 1940, it has increased from 85 to 165 or about 95%. This lack of correlation raises some questions as to the long-term value of this type of data. It should be pointed out, however, that costs estimates of this type are usually for a project rather than for one piece of equipment, and under these circumstances, variations tend to cancel.

Marshall and Stevens as well as other sources consider their index to be quite reliable for the industry as a whole. However, they do not consider it to be entirely reliable for any one particular piece of equipment.

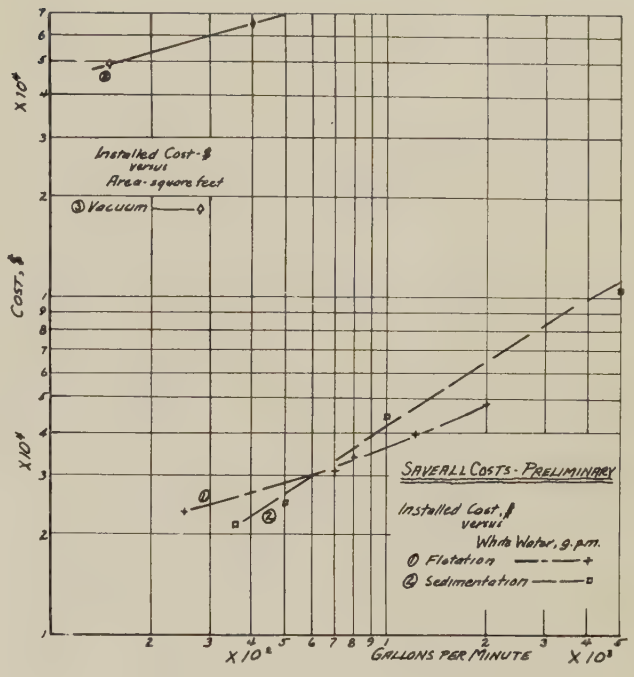
The failure of this item to follow the index is considered largely due to the fact that the application of this principle to savealls is relatively new. It is natural that construction, engineering, research, sales, and administration expenses are unduly high in the early stages of product development, and costs therefore take considerable time to become adjusted.

Flotation savealls were also investigated. Here, too, it was considered that capacity may be expressed in terms of size, within the limits of accuracy of this study. Again, however, it should be recognized that operating costs and efficiency will vary widely with conditions.

The installed costs of this type of saveall were computed over the range from 250 to 2000 g.p.m. in tile and corrosion-resistant materials excepting pumps and piping. These results also plot as a straight line on log-log scale, except that there is a break in the curve at the 800 g.p.m. level. Below this cost is a function of the 0.30 power of capacity and above, of the 0.41 power. Cost of the 250-g.p.m. unit was estimated to be 23,500 as against 50,000 for the 2000-g.p.m. size. It is not of too much significance to compare the changes in cost with an index over the past 10 years as design as well as the basis of sale has changed during this period. Purely as a matter of interest, however, the costs of the bare equipment were compared over this period, and it was found that the changes were not uniform with size. From 1941 to 1951, the 250-gallon unit increased 80%, the 700-gallon 63% and the 1200-gallon about 50% during which time Marshall and Stevens' index increased 95%. If cost figures had been assembled in 1941, their use at this date corrected for the index would have introduced errors of 9, 15, and 27%, respectively.

As stated above, however, such a comparison does not mean too much as the design changed during this period. The explanation for these variations is felt to be in the fact that prices were not stabilized during this period due to the relative recent development of the machine. The information on gravity-type savealls was to be prepared separately from this, but it is understood that this is not ready for presentation at this time. The feeling is, however, that the problems of cost estimation of this type would parallel those of the vacuum type. It is also expected that variations would follow the index better than the above types.

In each of the types of savealls considered here, prices reflect those of but one manufacturer. This is intended as a preliminary study to bring to the attention of the members of



TAPPI and this committee, some of the factors that are involved in a study of this type. It is felt that in most of these cases, prices are fairly competitive and that variations from manufacturer to manufacturer could be handled by either assigning limits or by plotting mean values.

It is emphasized again that this study is in no way intended as a basis for comparison of the various types of savealls. Rather it is an effort to determine the feasibility of a program on the part of TAPPI to assemble cost data on the major elements of pulp and papermaking equipment. Comments as to the practicability of assembling data of reasonable accuracy and as to the value of such data to the industry as a whole will be greatly appreciated.

It should be recognized that this represents a project of considerable magnitude and will require a great deal of time and effort on the part of the participating members. If the cooperation and help provided by the saveall manufacturers are any indication of that which might be expected from other suppliers, however, this work will be minimized.

LETTERS TO THE EDITOR

TAPPI Dirt Count Charts

To the Editor, *Tappi:*

From time to time in this mill we experience difficulties in the reproducibility of the TAPPI dirt count. To help check our testing personnel, I should like to make up a series of dirt sheets with a known number of dirt spots of various diameters. To do this, I should like information as to how or where the dirt spots printed on the dirt estimation chart were printed, and how the actual diameters were checked.

F. P. Moden, *Control Superintendent*
Anglo-Canadian Pulp & Paper Mills, Quebec, P. Q. Canada

To the Editor, *Tappi:*

The explanation of the original negative for the dirt estimation chart appeared in an article published in the *Paper Trade Journal* 114, No. 12: 61–62 (May 21, 1942) by Graff and Nihlin.

Prints from this negative are supplied to TAPPI by the Institute of Paper Chemistry. These prints, or rather a random sample from them, are checked for spot and bar areas by means of an eyepiece micrometer in a microscope. It is difficult to estimate the absolute border of an ink spot at higher magnifications but the areas are found to be well within the prescribed accuracy of 10%.

I. H. Isenberg, *Research Associate*
Institute of Paper Chemistry, Appleton, Wis.

Identification of L-*Rhamnose in Aspenwood*

To the Editor, *Tappi:*

Aspenwood consists of a complex mixture of carbohydrates and other materials. An estimate of the amount of the various constituents in the extractive-free wood can be made by employing the standard methods used in wood analysis.

Analysis of Trembling Aspenwood Meal[a]
(Oven-Dry Basis)

Lignin, %	16.4
Holocellulose, %	86.4
Alpha-cellulose, %	53.9
Pentosan[b], %	19.1
Uronic anhydride[c] (as $C_6H_8O_6$), %	4.7
Ash, %	0.4

[a] 40- to 60-mesh, alcohol- and water-extracted material.
[b] (Calculated as xylan and corrected for hydroxymethylfurfural and uronic anhydride.
[c] Calculated from the yield of carbon dioxide produced on boiling a sample with 12% hydrochloric acid saturated with sodium chloride.

It will be seen that the pentosan content is calculated as xylan. This calculation is dependent upon the assumption that other pentoses or methylpentoses are either absent or present in small amounts only, and that the uronic acid derivative present yields the same amount of furfural on distillation with 12% hydrochloric acid as does glucuronic acid.

We have hydrolyzed aspenwood meal with *N* sulphuric acid at 90° and have examined the mixture of sugars produced by means of the paper chromatographic procedure. This mixture was composed in the main of xylose but it also contained galactose and arabinose and traces of other sugars provisionally identified as glucose and mannose and the sugar acid glucurone. Other sugars and *L*-rhamnose were also present in small amounts. The latter was isolated as the crystalline hydrate after extraction of the appropriate sections of several paper chromatograms. The sugar showed $[\alpha]_D + 9°$ (HO, *c* 0.4), and formed a benzoylhydrazone melting at 188° and not depressed on admixture with an authentic specimen.

This is believed to be the first time that *L*-rhamnose has been isolated from an aspenwood hydrolyzate. It is present in small amount (less than 1%) and its function is unknown. The presence of this small amount of rhamnose will not vitiate the determination of the xylan content of the wood.

J. K. N. Jones, *Visiting Fellow*
from the University of Bristol, Bristol, England
J. R. Schoettler, *Member TAPPI; graduate student,*
The Institute of Paper Chemistry, Appleton, Wis.

Food Yeast from Sulphite Liquor

To the Editor, *Tappi:*

I was most interested in reading your very complete article entitled "Food Yeast from Sulphite Liquor," published in the October issue of *Tappi.*

On page 38A I noticed that the authors, describing the twin drum driers, indicated that the drums were 42 feet in diameter by 120 feet long. These would really be quite some drums to manufacture, ship or operate. We feel that you might be interested in correcting this to 42 inches in diameter by 120 inches long for the benefit of those who might not be quite as familiar with this type of drier.

There is another point which you might be interested in clarifying which has to do with the designation of these driers as "twin" rotary driers. Purely as a matter of differentiation in name our company considers a twin drum drier as one in which the drums rotate away from each other at top. The driers where the drums rotate toward each other at the top are designated as double drum driers. We believe that the driers at the Lake State Yeast Corp. are of the double drum variety. Although this is merely a matter of nomenclature, to those who are familiar with this differentiation between types of driers, it might clarify the description somewhat.

In general, however, we think that article was very well written and of considerable interest to the many people who have been following this whole subject.

W. D. Kohlins, *Manager, New York District*
Bufflovac Equipment Div., Blaw-Knox Co.

Physical Tests of Structural Type Boards

To the Editor, *Tappi:*

We have studied with considerable interest the Suggested Method T 1001 sm-51 for forming structural-type boards for physical tests of pulp. May we say first of all how much we appreciate the valuable activities of this committee and the advances which have been made in the development of standard methods for structural fibrous materials.

In connection with T 1001 may we suggest some minor modifications in connection with the standard board machine. Our experience over a number of years has been that there is a tendency to uneven forms of the board unless adequate space is left between the wire on which the sheet is formed and the drain. The standard board machine allows only 4 inches between these points and is connected direct to a 2-inch drain pipe. The board machine which we devised here a number of years ago has a drainage area beneath the deckle $7^1/_2$ inches in depth and the same width as the deckle. It then tapers in the form of a cone a further $7^1/_2$ inches deep to a 2-inch drain pipe. The objection in allowing this much volume of drainage is to avoid any tendency to nonuniform drainage through the wire.

We also prefer a circular board machine since in our experience the corners of a rectangular machine tend to permit thin spots to develop. There may, however, be good reasons in connection with the cutting of test samples for devising a board machine 14 by 14 inches.

If the committee wishes, we would be prepared to send forward a drawing of the unit designed and used in this laboratory. We do not consider this by any means perfect but there may be some points in its design which may be of assistance.

A. W. Mackney, *Chief Chemist*
N. Z. Forest Products Ltd., Auckland, N. Z.

Kraft Digester Corrosion

To the Editor, *Tappi:*

Last Summer we had the pleasure to see Douglas Jones, C.P.P.A., here in Stockholm, during his very short visit to Sweden. We had then the opportunity to discuss the sulphate digester corrosion problem briefly. Recently we got a letter from Dr. Thiesmeyer, Montreal, in which he told us about the Canadian committee and outlined the planned work. He assured us of their desire for close cooperation in these matters of mutual interest and believed that coordination in research begins at the planning stage, and does not consist simply of an exchange of technical reports after the work has been done.

Our corrosion committee discussed these matters at a meeting, October 26, and decided to send information to the TAPPI Digester Corrosion Subcommittee and to the corresponding Canadian committee regarding the membership of our committee, our work program, and the preliminary results, which we so far have gotten.

Previously a subcommittee to our sulphate committee has been studying the corrosion problems in sulphate mills. The result was published by C.-G. von Essen in *Svensk Papperstidn* 52: 549 (1949).

The representatives for the sulphate industry considered, however, that the work on these problems had to be continued and more intensified. Thus in the beginning of this year a special sulphate corrosion committee was appointed with N. Wegner as Chairman. This committee, which is a technical advisory committee, has the following members:

N. Wegner, Fredriksberg, *Chairman*
G. Edling, Stockholm
C.-G. von Essen, Väja,-Dynäs
C. G. Geijer, Åmotfors
R. Hellerqvist, Östrand
E. Hägglund, Stockholm
E. Rudberg, Stockholm
C.-U. Willers, Strömsnäsbruk
L. Stockman, Stockholm, *Secretary.*

Dr. Hägglund is Director of Research at this Institute; Dr. Rudberg is Director of the Metallurgical Institute, and Dr. Edling is in charge of one department of the Swedish Steam Users Association. The other menbers with exception of the Secretary are representatives for the sulphate industry, especially from sulphate mills with bad digester corrosion.

There are also four associate members from two iron works, Avesta and Uddeholm, and also from two manufacturers of arc welding electrodes, ESAB and ASEA.

The main problem, which had to be attacked immediately, was that of sulphate digester corrosion and the work program was therefore concentrated on this matter. The experiments are carried out here at the laboratory in close contact with the industry. So far, the personnel is very limited, being only two people with the secretary as supervisor.

We wished to start the work without any preconceived theories, except the fact that we knew from earlier investigations made here at the laboratory, that the concentration of sodium sulphide definitely has an effect on the rate of corrosion. The increase of the sulphidity during the last years cannot, however, explain the tremendous increase in corrosion, so we decided to try to find out the influence of various components of the white liquor on the rate of corrosion. On the other hand, there are many indications that the steel composition of the digester shell also plays an important role. The work program also includes therefore experiments on steels with various concentrations of Si, C, and Mn.

In order to determine whether the white liquor composition had any effect or not, we made corrosion tests using white liquors from eight different mills. The concentration of sodium sulphide was corrected and made equal in all the liquors in order to eliminate the influence of the sulphide. The uncorrected composition is given in Table I. It will also be seen from the table that the rate of corrosion is varying within a very wide range. It may be mentioned that mill A is the Swedish sulphate mill with the most severe corrosion problems. The opposite is true for mills B and F.

Table II

$Na_2S_2O_3$, g./l.	Corrosion per cook, g./m.²
0.0	8.1
2.7	15.4
5.0	21.8
10.0	23.5
15.0	24.0
Addition of sulphur, 0.5 g./l.	29.2
Without addition	20.8

Although the variables in the liquor composition are many, we found it probable after having studied Table I, that the thiosulphate content could have some effect. In a separate run we varied therefore only the concentration of this substance. The result may be seen in Table II, which also includes a test without and one with an addition of 0.5 g. of sulphur per liter.

It is obvious that the thiosulphate and sulphur and possibly also other constituents of the white liquor will have a pronounced influence on the rate of corrosion. The work on this and also on steel composition will continue.

It is our desire that the work on these matters in Canada, U.S.A., and Sweden will be continued in closer cooperation.

I hope that I will have the opportunity to discuss these things with you personally later on. I expect to arrive in the States near the end of January, 1952.

LENNART STOCKMAN, *Swedish Forest Products Research Laboratory, Stockholm, Sweden*

Cuprammonium Disperse Viscosity Test

To the Editor, *Tappi:*

You probably are familiar with the fact that for some years one of our standard offerings to the pulp mills has been a group of four viscosity reference oils used for calibration of viscosity tubes in the cuprammonium disperse viscosity test of pulp.

These offerings were originally made some years ago after we had been requested to furnish such a need of the mills by several of those prominent in your membership. Following our announcement of their availability, an editorial notation appeared in your Journal advising the membership where they might obtain those oils.

A number of years have passed since the occurrence of our first offering and we have never found the business of supplying these oils highly profitable because of the fact that so few of the mills find occasion to use them. It is true that we do receive occasional orders, but it is rare in any one year that we sell more than 20 to 30 quarts among the several types offered.

It would have been our purpose to continue this service at very close to the original price designated some years ago, but we recently have been faced with very heavy cost increases and, unless those increases may be borne in a new price structure and may prove of commensurate value to the small group of those who have employed our standards, we likely will be

Table I

White liquor	Grams per liter							
	A	B	C	D	E	F	G	H
Na_2S	42.1	37.8	33.3	43.6	42.8	25.7	50.7	33.8
NaOH	109.2	100.4	98.5	96.8	94.4	86.0	98.8	103.6
Na_2CO_3	46.1	31.3	30.0	41.6	42.9	31.2	34.3	28.9
Na_2SO_4	17.6	17.6	17.4	18.7	20.19	18.2	16.8	17.6
Na_2SO_3	0.76	1.58	0.00	0.01	0.69	0.34	1.67	1.20
NaCl	3.28	5.15	3.74	2.89	3.60	4.29	5.69	3.51
SiO_2	0.23	0.11	0.21	0.24	0.14	0.02	0.06	
$Na_2S_2O_3$	10.28	7.61	5.91	3.42	2.74	2.33	7.15	8.87
Fe_2O_3 + Al_2O_3	0.22	0.26	0 0.50	0.01	0.21	0.49	0.07	0.31
Corrosion, g./m.², day	47.6	10.3	21.6	24.8	23.8	7.1	13.0	27.9

obliged to discontinue their supply. Before reaching a decision in the matter, I have felt it well to draw this to your attention and ask if you will either advise us directly from your office or submit the question to those in the organization who would most likely have occasion to desired continued availability of those standards.

MILTON I. HARTMAN, *President Hartman-Leddon Co., Philadelphia, Pa.*

To the Editor, *Tappi:*

Milton I. Hartman of the Hartman-Leddon Co. wrote in with regard to four viscosity reference oils used for the calibration of viscosity tubes in the cuprammonium disperse viscosity testing of pulp.

We have a subcommittee of TAPPI now working on the revision of viscosity methods and, as far as the official recommendations of the new standard are concerned, they are not likely to contain a reference to the oils referred to by Mr. Hartman. However, they may still find use by various mills as a practical standard for their viscosity work. The type of viscometer which will be finally recommended by the committee is now under discussion and we cannot make a decision until a research project has been completed at the Paper Institute. However, it seems likely that when the work is completed the standard will recommend the use of a Bureau of Standards reference oil for the checking of the instrument.

The price increase which was suggested in Mr. Hartman's inquiry seems to be a logical solution to the present problem. The oils will undoubtedly continue to be used at the same rate as they are now despite any price increases.

In conclusion, we would say that the official TAPPI standard is not apt to go back to reference oils other than those from the Bureau of Standards. However, mill methods and control methods may rely upon secondary standards.

A. S. O'BRIEN, *Chairman*
Chemical Methods Committee

To the Editor, *Tappi:*

In reference to the letter from the Hartman-Leddon Co. it would appear from the note in the third column of T 206 m-44, Cuprammonium Disperse Viscosity of Pulp that arrangements were made with H.-L. Co. to furnish mineral oil reference standards for calibration. This method is now under the jurisdiction of the Chemical Methods Committee although it was probably originally set up by the Pulp Testing Committee. I believe it has been under consideration for revision for several years, but no action as yet.

In any event I should think the H.-L. Co. is entitled to charge whatever they consider to be a fair price for the calibrating oils, and if there is not sufficient demand, to discontinue them. This would not be fatal to the method, as the preferred procedure of calibration (as stated in the method) is with glycerine. The oils are merely more convenient.

ROGER C. GRIFFIN, *Chairman*
TAPPI Standards Committee

To the Editor, *Tappi:*

I thank you for your letter with its various enclosures.

Continued sale of the oils involved in the viscosity measurement has not been a very important factor from our standpoint. I probably mentioned in my previous letter that the annual volume requirement was far less than originally anticipated.

In view of that condition as well as the statements of the enclosures with your letter, we most certainly shall not plan to go further in the matter upon the basis of the recently highly advanced costs. We shall plan to continue making available three of the four offerings which are still in our stock. Likewise, upon the occasion of any future orders for

View of new additions, Fuji Mill, Honshu Paper Co.

the one type of oil which would require replacement were we to continue its supply, we shall write to each such mill attempting to place an order with us and shall inform it of the current status and the likely future status of withdrawing all offerings of these oils. If you feel it desirable to inform your membership through the editorial columns of the Journal, there certainly would be no objection to your so doing insofar as our interests are concerned.

MILTON I. HARTMAN, *President*
Hartman-Leddon Co.

Honshu Paper Mfg. Co. Developments

To the Editor, *Tappi*:

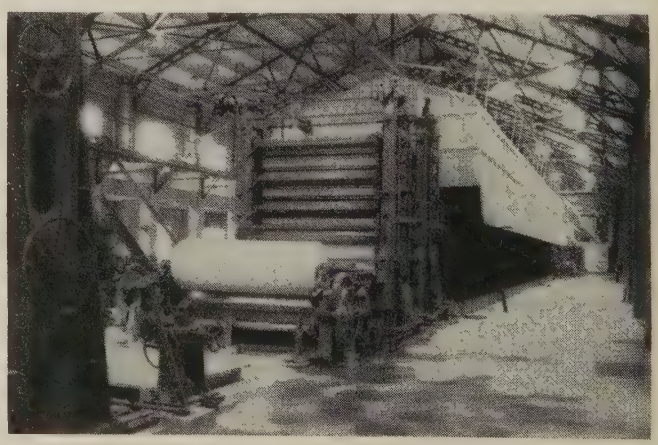
I wish to thank you personally and sincerely for the valuable information you have been sending us from time to time. Your periodical publications are found very useful not only for our technical men, but also for us management people.

Recently so many Japanese chemists and engineers who are engaged in the Japanese paper and pulp industry have visited your country. They have brought home with them your up-to-date know-how and some of these technical improvements have already been adopted in our mills.

However, on account of linguistic handicap as well as of conservative characteristic of Japanese people, our technicians are very slow in responding to your call (dated July 27, 1951) in sending in their reports on their findings.

Honshu Paper Co. has completed its one-year expansion program at Fuji Mill by the end of last August. Two 15-ton digesters and one 102-inch fourdrinier paper machine and four 10-ton B. & W. boilers, all of domestic design and manufacture, are installed, resulting in a monthly production increase of 2,000,000 lb. of paper and 1500 tons of sulphite pulp.

YOSHIO ICHIKAWA, *Director*
Honshu Paper Mfg. Co., Ltd.



Machine at Fuji Mill, Honshu Paper Co.

LOCAL SECTION ACTIVITIES

Reports of Meetings, Personnel, and Events

Chicago

The Chicago Section on November 19 joined with the Folding Paper Box Association of America in conducting an afternoon and evening meeting of folding cartons, with particular attention to greaseproof boards, papers, coatings, films, and foils. The meeting synchronized with the regular monthly meeting of the Chicago Section of TAPPI and was held at the Chicago Bar Association Headquarters, Chicago, Ill. Well over 200 were in attendance, with folding carton men from as far west as the Pacific Coast and as far south as Louisiana attending. Program arrangements were under the direction of W. D. Hall, Research and Technical Director of the Folding Paper Box Association, and of Peter J. Trucano, Chief Chemist of Morris Paper Mills, as Program Chairman of the Section.

Vance V. Vallandigham of Kelco Co., as Chairman of the TAPPI Chicago Section, presided, and F. D. Long, Technical Director of Container Corp. of America and Chairman of the Research and Technical Committee of the Folding Paper Box Association, was moderator.

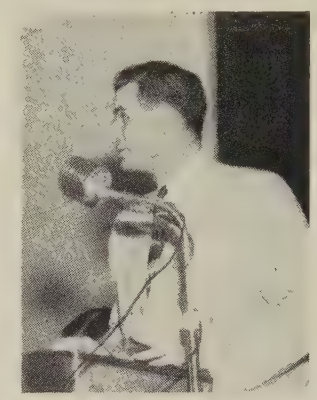
In opening the session as moderator, Mr. Long said that perhaps the approach of seeking a perfect material from the greaseproof standpoint was a mistake. Instead, it might be best to view the problem from the standpoint of actual use, where sometimes protection is required for only a few days, as contrasted with other instances where longer protection is required. This opens the way for many papers, coatings, films, and other materials and processes. He pointed out that the all-purpose grease-resistant material might well be too expensive for consumer packaging. He then introduced the speakers, who, together with their subjects, were as follows:

Polyethylene Coatings, by L. E. Whitmore, Bakelite Co., Chicago, Ill.
Greaseproof Papers and Laminations, by M. L. Downs, Thilmany Pulp & Paper Co., Kaukauna, Wis.
Aluminum Foil and Paper Laminations in Packaging, by A. I. Totten, Jr., Director of Research, Reynolds Metals Co., Richmond, Va.
Special Greaseproof Method and Formulation, by Dr. D. R. Erickson, Consultant, Kalamazoo, Mich.
Survey of Greaseproof Boards and Papers, by A. T. Luey, Sutherland Paper Co., Kalamazoo, Mich.
Greaseproof Requirements (the consumer viewpoint), by H. T. Spannuth, Wilson & Co., Inc., Chicago, Ill.

Group at the November meeting of the Chicago Section

Polyethylene Coatings. Mr. Whitmore mentioned the general characteristics of polyethylene as including relatively high gas transmission rate, but relatively low moisture transmission rate, affording good properties for packaging fruits and vegetables, where escapage of gas may be advantageous. Since 1945, he said, polyethylene resins have found wide acceptance in packaging either as modifiers for paraffin wax, as unsupported thin films, or as thin films laminated to papers or boards. Grades range in molecular weight from approximately 7000 to as high as 24,000 commercially, with small samples of higher and lower grades available for development work.

Its chemical inert nature makes it difficult to compound with other materials and it is not wholly compatible with itself. It is most closely compatible with paraffin wax. However, polyethylene modified wax mixtures may be effected using conventional equipment, without major alteration. This combination adds to flexibility and the result has some of the properties of both materials.



F. D. Long, Container Corp. of America

Polyethylene coatings on board or paper are applied either by a hot melt method or an extrusion lamination method. The extrusion lamination method involves, first, the extrusion of a film, and second, the immediate lamination to a web of board or paper. In certain cases, a third method involving application from hot solution is being used. For this purpose, polyethylene, Mr. Whitmore said, is soluble in commercial solvents, including toluene, at a temperature of about 90°C. With either method, it was found that higher molecular weights of polymers slowed operations so that lower molecular weights have been used with best results.

Mr. Whitmore said that polyethylene was not the answer to all problems of producing greaseproofness; that its greaseproofness was relative; but that the material took a long step toward a completely satisfactory material. He believed that the vinyls have a very definite place in the greaseproof picture. Yet, he felt that polyethylene would open a whole new field of applications. He believed that polyethylene and the vinyls would finally settle down to certain definite applications.

In closing, he said that polyethylene as a paper or board coating makes possible the production of packages with physical characteristics not heretofore possible with any previously known coating material. Particular advantages when used in conjunction with wax, he mentioned as: (1) resistance to grease transfer before and after conversion, (2) good moisture-vapor-transfer resistance, (3) good heat sealing properties, and (4) good flexibility.

Greaseproof Papers and Laminations. Mr. Downs said that, with more than one method of solving the greaseproof problem, glassine and greaseproof papers provide some of the less costly methods of attaining the goal, because these sheets can frequently do the required job for 30 to 50% of the cost of competitive materials. The results are not as absolute

L. E. Whitmore, Bakelite Corp.

M. L. Downs, Thilmany Pulp & Paper Co.

as might be obtained with some other materials but he pointed out that the cost of materials that would give absolute protection frequently are too costly for the product in question.

Both glassine and greaseproof papers were described by Mr. Downs as made from pulp (sulphite or kraft) that is treated to result in high pentosan and low alkali-cellulose content. Both are given long beating time, particularly in connection with glassine so that the freeness is very low and the result is more or less a cellulose jell. The result is a slow-running sheet with very close formation and, although a standard fourdrinier is used, the wet end is modified to get faster drainage. The foregoing would result in a greaseproof sheet and, if glassine were desired, the sheet would be remoistened and then supercalendered with pressures of 1500 pounds using a stack of steel and cotton rolls. Thus, glassine is merely a transparent form of greaseproof paper although it is more greaseproof because it is more compact. With glassine, grease transfer is mainly through pinholes or through capillarity at points of raised fibers.

Mr. Downs explained that the turpentine test for evaluating greaseproofness was developed because it was a convenient test that could be made in the mill by operating men. While the test does not exactly parallel the greaseproofness of a sheet, it gives a quick and good indication. Of course, he pointed out, the turpentine test should not be used for sheets which are soluble in turpentine.

Commonly used glassine and greaseproof sheets in the board industry are 25–30 pounds basis (24×36—500), which are readily available, with other weights also available. Many of the sheets are opaqued with titanium dioxide to cover up the chipboard to which the greaseproof sheet may be laminated. These papers are also frequentyl used in colored form on folding cartons or in corrugated forms for bakery goods. In laminating to board, aqueous adhesives are not ordinarily used because they cause glassine and greaseproof to swell and to reverse the properties, but aqueous adhesives in modified form (possibly of higher viscosity) can be used.

Frequently, also, Mr. Downs said, micro waxes are applied to the films to lend moisture-vapor resistance or other properties. Furthermore, because of the hardness and evenness of caliper of glassine, it is sometimes used in lamination with board where it is desired to achieve an even wax barrier on the board, even though glassine itself might not otherwise be indicated. In laminations, different adhesives (dextrines or waxes) are used, depending upon the properties desired in the finished board or box.

Other paperboard container uses, where the glassine or greaseproof paper is used in the form of inner bags, laminations to the board, outer wraps, etc., were mentioned by Mr. Downs as cheese, baked goods, meat products, and many others and it is also used where a "pick-free" surface is desired.

Aluminum Foil and Paper Laminations in Packaging. Mr. Totten said that, while but a small portion of aluminum is devoted to foil-paper laminants, the aluminum value for this purpose represents a market of about $15,000,000 and 70% of the foil is laminated to board and paper. The lamination is usually due to the fact that the foil is delicate and needs the support of the paper at the same time that the lamination makes possible use of a thinner foil. An example is the widespread use of foil and glassine or greaseproof paper for wrapping margarine and butter, using thermoplastic adhesive. He said that margarine people will conserve about 10 million pounds of product per year, product that would otherwise spoil, by using this wrap.

In connection with the paperboard containers, Mr. Totten said that there are many applications possible in combination with foil combinations. One mentioned was a bag or liner of two sheets of foil laminated to a thin sheet or paper, resulting in high resistance to moisture-vapor transfer. Another was the use of foil or a lamination as the outer wrap of a fiber can, which not only results in high MVT resistance, but greatly improves the appearance from a marketing standpoint. Foil or its laminants can also be used as carton inner- or overwraps and common examples mentioned were dried fruits and the overseas shipment of cartons of cigarettes. Foil wrapped cartons for photographic film shipped overseas have so added to protection, Mr. Totten said, that they have eliminated need for imprinting the photographic film expiration date. He also mentioned possibilities of vinyl and foil laminations for grease resistance.

Particularly in connection with folding cartons, Mr. Totten said that overwraps and laminations to the board itself have materially promoted folding cartons, opening new fields for use. Many of the advantages are decorative with aqueous adhesives used for lamination. On the other hand, wax laminations furnish additional MVT resistance because the wax fills up any pinholes there may be in the aluminum. Protective packaging jobs of foil and cartons mentioned were cigarettes, ball bearings, and cartons for mailing matches.

Mr. Totten felt that there was room for further development, as a part of the carton or as inner and outer liners, despite the fact that aluminum is a short material, as evidenced by the fact that it is subject to the Controlled Materials Plan. However, he thought it obvious that if a carton using aluminum could be developed that would save large quantities of metals in the form of another container, then the Government would look with favor upon allowing the small quantity of aluminum that might be required for the new development. He also felt that many such developments would remain in use after the present emergency.

He mentioned as reasons why aluminum fits in well with folding cartons the facts that aluminum is relatively free of pinholes, it can be bent sharply (when annealed) without fracturing, it is nontoxic, and it has high resistance to grease

A. I. Totten, Jr., Reynolds Metal Co.

D. R. Erickson, Consultant

H. T. Spannuth, Wilson & Co.

A. T. Luey, Sutherland Paper Co.

and moisture-vapor transfer. He believed that the full possibilities have not been exploited, and he said that the aluminum industry would be glad to cooperate in such developments.

Special Greaseproof Method and Formulation. Dr. Erickson said it has long been known that many polymers, if they could be placed in a continuous film on board or paper, would result in complete greaseproofness. The difficulty has been that the porous nature of the sheets has resulted in drawing the solvents and polymers into the sheet, resulting in imperfect films. Possibilities mentioned were plasticized resins for hot melt applications and polyethylene. However, most of Dr. Erickson's paper was devoted to a new method of greaseproofing by precipitating a polymer that is insoluble in water and preferably in other solvents in such a manner so that the polymer remains on top of the sheet in a continuous film. He said that alkali soluble hydroxyethyl cellulose seems to be such a material; it is soluble only in dilute aqueous sodium hydroxide and can be precipitated from its solution and neutralized by contacting with any aqueous acid salt or ammonium salt solution. By this means, the solution is transformed to a continuous solid film and the contained water is simultaneously released as free water. The water can then be squeezed and dried out in the normal manner.

This is referred to as the precipitation method of coating, resulting in films of about 0.001. The resulting board or paper stock is similar to one laminated with a thin sheet of P.T. Cellophane, having resistance to grease, oil, and solvents and presenting an excellent barrier to oxygen and CO_2 transmission. Low temperature flexibility and MVT resistance were described as no better than for cellophane so that thin films of lacquer or resin should be applied for high MVT resistance and these films may be as low as $^1/_2$ pound per M square feet because the solvents cannot penetrate the Hydroxyethyl Cellulose (H.E.C.) surface.

Moreover, Dr. Erickson reported, concentrations up to 80% clay or pigments do not seem to affect H.E.C.'s properties as barrier to oils, waxes, and solvents. Also, the highly pigmented coatings have good covering properties, resulting in surfaces with remarkable printing qualities, even for letterpress. The printing and adhesive problems with such materials are similar to those with cellophane; that is, if the coating is pigmented, there is little problem with adhesives or printing and, if it is not pigmented, the problems are the same as for cellophane. The reverse roll type of coater has proved most satisfactory for this type of coating, Dr. Erickson said.

Survey of Greaseproof Papers. Mr. Luey gave a classification of grease resistance by end uses; covered methods of testing or evaluating grease resistance; listed materials available or in use at present; and presented the general cost picture in connection with different materials.

Greaseproof Requirements. Mr. Spannuth said that greaseproofness in packaging should signify more than protection of appearance from migration of the fatty substance. It should mean economical, convenient, and attractive packaging so that the qualities of the product are adequately protected as related particularly to the fatty ingredient. This should also be achieved with proper perspective to other requirements for good packaging.

This may call at times, he said, for material free of prooxidant effect or even give antioxidant effect and other requirements might be (1) barrier to oxygen or odors, (2) possibly permit passage of odor, (3) opacity to light or permit passage of certain wave lengths, and (4) give support, insulate, or conceal limited soakage. He then gave some aspects of fat deterioration, covering the effects of oxidation, certain printing inks, absorption of odors, rust in metallic items in packaging, and many other critical conditions that arise in the packaging of fatty items.

WARREN R. PRICE, *Publicity Chairman*
Chicago Section

Lake States

The second Lake States TAPPI meeting of the 1951–52 season was held in Wisconsin Rapids, Wis., on the afternoon and evening of November 15. The formal meetings were preceded by a well-organized visit through the semichemical plant, the sulphite mill, the bleach plant, and the paper mill of Consolidated Water Power and Paper Co., Wisconsin Rapids Division. An afternoon panel discussion on Hardwood Utilization was held at the Elks Club, Wisconsin Rapids, with C. R. Faulkender, Technical Director of Hoberg Paper Mills, in charge of the program. This was followed by a dinner at 6:30 and a talk by M. N. Taylor, Executive Director of Trees for Tomorrow, on the over-all objectives of that organization. Abstracts of the papers are as follows:

Paul West, Pulp Mill Superintendent, Thilmany Pulp and Paper Co., Kaukauna, Wis., discussed Pulping of Hardwoods by Modified Soda Process.

Thilmany has pulped hardwood by the kraft process, but kraft hardwoods, especially poplar, are not too good for their particular papers. Around 4% of their wood consumption is poplar, which they have used straight or mixed up to 20% of the wood furnish in regular kraft cooks. Poplar kraft did improve the mullen, made beating easier, and sometimes did

A. P. Adrian, Neenah Paper Co.; C. R. Faulkender, Hoberg Paper Mills; H. W. Rowe, Nekoosa-Edwards Paper Co.; and G. K. Dickerman, Consolidated Water Power and Paper Co.

not hurt the tearing strength of the finished paper because of the easier beating.

In 1945–46 they needed some soda pulp and tried bleaching kraft poplar with conventional 2-stage bleach. Results of this 2-stage bleaching were not too good. In 1947–48 they cooked some poplar with NaOH only and returned the spent liquors to their regular kraft recovery. Commercial 50% caustic was placed in regular white liquor storage, from which it went to the measuring system where it was diluted with water and sent to the digester. It was calcualted that approximately 80% of the NaOH charge was returned to the kraft recovery.

They found that the modified soda poplar pulp was much easier to wash and handle than regular kraft poplar, permitting them to make around 50 tons per day of bleached soda poplar as compared with only 25 of bleached kraft poplar. The soda pulp was better for soft, bulky sheets.

Pulping and bleaching data on Thilmany's bleached poplar kraft compared with their bleached poplar soda are given in Table I.

Table I

	Bleached poplar kraft	*Bleached poplar soda*	*Soda + or − %*
Pulping:			
Wood, cords/digester	8.5	8.5	
Chemical			
Total volume, gal./digester	4000	5000	+25
White liq. or 50% NaOH, gal./digester	2600	800	
Black liq. or water, gal./digester	1400	4200	
Active Na_2O, lb./digester	2600	3900	+50
Steaming			
Pressure, p.s.i.	110	110	
Time, min.	100	100	
Permanganate No.			
Blow	15.5	11.5	
Wet machine	11.5	10.0	
Yield			
Tons/digester	4.50	3.75	−18
Active Na_2O, lb./ton	580	1040	+80
Bleaching:			
1st Stage			
Cl_2/ton	165	...	
% Cl_2	8.25	...	
Time, min.	57	...	
2nd Stage			
Cl_2/ton (hypochlorite)	25	95	
% Cl_2	1.25	4.75	
Time, min.	120	105	
Temperature, °F.	100	100	
Total % Cl_2	9.50	4.75	−50
G.E. brightness	68	73	+ 5

Hugh Miller, Pulp Mill Superintendent, Green Bay Paper and Pulp Co., Green Bay, Wis., discussed Semichemical Pulping of Hardwood. This paper will be published later.

Henry Grabowski, Sulphite Superintendent, Falls Power and Paper Co. (Division of Scott Paper Co.), Oconto Falls, Wis., discussed Sulphite Pulping of Hardwoods.

Hardwood Sulphite Pulping

In the preparation for this paper, Mr. Grabowski sent questionnaires to all the sulphite mills in Wisconsin asking for their production figures on hardwood sulphite per year from 1946 through 1950 as compared with their total unbleached sulphite production. The replies from 11 different mills in Wisconsin showed that hardwood sulphite constituted 15.9% of the total unbleached sulphite production in 1946 and has risen to 22.2% in 1950. A total of around 450,000 tons per year of hardwood sulphite is now produced by these Wisconsin mills.

At the Falls Paper and Power Co., the total capacity of their mill is around 90 tons per day of hardwood compared with 70 tons per day of softwood sulphite. This is chiefly due to the higher yields of hardwood, but is also influenced by screening and other problems peculiar to their equipment.

Six out of the 11 mills replying cooked their hardwood separately. Among the advantages for cooking separately are the following:

1. Hardwood can be pulped at a maximum temperature about 10°C. lower than softwood sulphite. In a mixed charge, there is the possibility of overcooking the hardwood and undercooking the softwood. This leads to the possible serious economic loss of valuable softwood fibers in the screenings.
2. By cooking hardwood separately, there is more flexibility in the percentage of hardwood sulphite to be used in furnishes for various grades of paper.

Against cooking separately are the following:

1. If slush systems are used, large storage capacity is required for separate cooks of hardwood and softwood sulphite.
2. If the pulp mill capacity is smaller than that of the paper mill, the hardwood and softwood can be cooked together and various furnishes in the paper mill can be arranged by adding various amounts of purchased pulps, either hardwood or softwood as desired.

Mr. Grabowski presented some of their operating experiences with hardwood sulphite at the Falls mill. Hardwood, especially poplar, is not as scarce as softwood such as spruce. Rough poplar is quite available. There is some peeled poplar but it still may need considerable cleaning. They experienced about 11% shrinkage in running rough poplar through their barker and may have to cook a bit more drastic to take care of some bark that is not removed. However, the wood room capacity is higher for hardwood than softwood.

They find that hardwood chips catch fire more easily in the bins than softwoods. This is aggravated by the fact that they use superheated steam for dislodging chips. With this procedure, they can store only one half as much poplar chips as spruce chips in a bin.

For cooking poplar by the sulphite process, they use chip packing, forced circulation, hot acid system, and indirect cooking. A typical schedule is 3 hours to 135°C. (90 p.s.i.) and a total cooking time of 8 hours (9 hours cover to cover). Acid concentration is 6% total of 1.1–1.2 combined. With this schedule, they obtain a permanganate number of 9.0.

With hardwood sulphite they experience considerable foaming on the decker.

In bleaching, they must have good control of pH and temperature in order to maintain strength. They are shooting for a pH of 7.0 in the finished pulp with a maximum temperature of 39°C. in the low density hypo stage.

They have found that unbleached poplar sulphite pulp, especially when fresh, changes color rather easily. The addition of alum at the beaters may turn the pulp quite red or contamination from lime dust may send it to the green side. Aging of this pulp helps color stability.

They have used up 85% bleached poplar sulphite in a toweling furnish and up to 75% in a tissue furnish.

George J. Brabender, Assistant to Vice-President for Research, Marathon Corp., Rothschild, Wis., discussed Cooking and Bleaching of Hardwood Kraft.

Marathon Corp. cooks hardwood only in its Canadian mill, where they have found their timber rights contain about 50% hardwood, one half of which is aspen. This aspen is larger and of higher density than our aspen and is obtained in high-grade, relatively pure stands. They are cooking hardwood mixed with softwoods because it is very difficult to wash a straight hardwood cook on their brown stock washer. They seldom cook less than 20–25% softwood in their "essentially hardwood" cooks for this reason.

Marathon has found that the strength of their bleached poplar kraft from the aspen in their Canadian holdings approaches the strength of bleached hemlock sulphite from this area. Some typical pulping data on hardwood kraft (essentially aspen) compared with softwood kraft (mixture of spruce, balsam, and jack pine) are as follows:

Table II. Marathon of Canada

	Hardwood	*Softwood*
Active alkali	13%	18%
$KMnO_4$ No. of pulp	11–12	17–18
Yield by weight	53–54%	43–44%
Wood density	23 lb./cu. ft.	27–28 lb./cu. ft.

The yield by weight from hardwood is considerably higher than it is from softwood. However, the lower density of the hardwood makes the yield per cord correspondingly lower for aspen.

Balm of Gilead does not give as high quality pulp as aspen, although the yield is about the same; the fibers are shorter.

Marathon has not cooked birch in its Canadian mill, because the high density of this wood when wet gives excessive sinkage on river driving. They may bring some in by truck after their permanent logging camps are established. Birch is hard to bark but gives high quality and yield. Density is high at about 31 pounds per cubic foot and gives yields in the neighborhood of 53–54% by weight. For a given amount of pulp, the amount of wood required would be in the ratio of 1.4 cords of birch to 1.8 cords of aspen, spruce, or jack pine. Birch should be OK for hardwood pulping if the above difficulties can be overcome.

One very important difficulty in cooking hardwoods by the kraft process is their effect on liquor recovery. The fact that hardwood gives better yield means that there will be lower dissolved solids in the liquor sent to recovery (approximately 0.8 pounds dissolved solids per pound of pulp for hardwood compared to 1.3 pounds of solids per pound of pulp for softwoods). This means that there is about 50% more dissolved solids from softwood than hardwood, which makes it difficult to keep the fires in the recovery furnace going on long runs of hardwood.

A typical Marathon hardwood kraft is cooked at a maximum temperature of 170°C. (340°F.) for 3 hours to $3^1/_4$ hours. This pulp is then bleached in an 8-stage bleach plant to a brightness of 82–84%. Bleaching is satisfactory if 20–25% softwoods are in the pulp to facilitate washing, etc. Cleanliness of this pulp is satisfactory.

It handles satisfactorily over their pulp driers as long as 25% softwood is present.

C. K. Textor, in charge of Pulp and Paper Division, Bauer Brothers Co., Springfield, Ohio, discussed Development in the Refining of Hardwoods.

This paper will be published later.

Trees for Tomorrow was the topic of M. N. Taylor, Executive Director of Trees for Tomorrow, Inc., at the dinner meeting.

Mr. Taylor's remarks follow:

"I am indeed happy to appear before this topflight group of technical men from the paper industry of the Lake States and report on the semipublic program of Trees for Tomorrow, Inc.

"During the past eight years this program has been directed toward laying down a workable pattern whereby industry could project good forestry among private landowners, schools, clubs, and entire communities, without duplicating what is already being done in this field by public agencies.

"Because you deal with facts and figures, and because you have more than an academic interest in forestry, I felt you would be primarily concerned with techniques applied to accomplish our objectives.

"The subject of your seminar today was hardwood utilization. The topic itself points up the giant strides that have been made since the time timber was considered a single-purpose raw material for the production of lumber. These strides establish a hypothetical slide rule with which to calculate the economic future of the Lake States.

"We have the land, millions of acres in Wisconsin alone, which are best suited for the growing of trees. We have a ready market for the sale of wood in Wisconsin's 4 million dollar a year paper industry. We have water and power....we have the research facilities of individual mills, as well as those of the Institute of Paper Chemistry and the Forest Products Laboratories...but we fall short in a near-by, dependable source of supply of wood. A recent survey by the Lake States Forest Experiment Station reveals that pulpwood production for the Lake States amounted to 1,872,000 cords in 1950. Of the regional total, Wisconsin produced only 25%.

"That is why common sense dictates that it is just good business to maintain a pace in forestry that matches the steady progress of science in our laboratories.

"And good progress is being made, especially during the past five years. More and more people no longer consider forestry a crusade, a visionary something, or a fantastic dream. In the same way public apathy toward sound conservation practices is giving way to action programs.

"In fact, never before in the history of this nation has so much interest been shown on the part of the general public in forestry. This interest stems to a great extent from the fact that people are resource conscious. At this moment we are not in a total fighting war with Russia, but we are engaged in a total and bare knuckle economic war with communism—and economic wars are won with resources. How we use and how we replace our renewable natural resources is a matter of prime concern to the over-all economy of this country.

"But naturally within this framework of keyed-up interest we have many varied and specialized interests. To some, the deer problem and whether we should have an open or closed season is of paramount importance. To others, the number and species of fish in our lakes and streams, pheasant rearing, duck population trends, or perhaps stream pollution are absorbing topics of discussion and action.

"If these specialized interests are misdirected, if too much emphasis is placed on one to the detriment of others, then the entire idea of good resource management can be thrown out of gear. However, when impetus is given to good counsel and planned effort much is accomplished. Good resource management is not based solely on forestry, or on soil, water, recreation, or wildlife management. There is a closely knit relationship between all of these factors, and each is dependent upon the other.

"The same comparison can be drawn within industry. Management cannot put all its emphasis on forestry. It must maintain a balance between the production of timber, manufacturing, research, labor relations, and of course, sales. A weak link in one of the many steps which converts the cellulose of wood into paper results in loss of sales, quality of product, and ultimate profits.

"When industry talks and acts in terms of long-range resource management, in addition to a future supply of timber, it stimulates the interest, it receives the cooperation and the active support of the public. This support is essential because pulp mills are dependent for their raw materials upon a steady flow of forest products.

"The above is not altruistic thinking, but constructive, sound business judgment when consideration is given to the sources of additional supplies of pulpwood in the state.

"Today Wisconsin is recognized as a top ranking state in industrial forestry, with 606,000 acres under management. However, if the wood-using industries of Wisconsin obtained the highest yield from their own forest land, there would still be a wide gap between the requirements of these industries for the raw material wood and the supply available from their own forest land.

"The largest classification of ownership of forest land in Wisconsin is not owned by industry. It is not classified as national, state, or county forests. The single largest classi-

fication of commercial forest land is controlled by private landowners who own 68% of the forest land in our state.

"It is recognized that good forest practices are being carried out on the remaining 32% of the forest land. The urgent need is to project good forestry practices to the remaining 68% of Wisconsin's woodlands.

"That is why in February of 1944 Trees for Tomorrow, Inc., was organized by the Wisconsin Valley papermaking industries. Originally six counties were set aside where an intensive field program could be projected. Since then this territory has been expanded to eight counties.

"This program is divided into two phases and deals with (1) the immediate present, and (2) the future.

"Under the heading of 'immediate present' Trees for Tomorrow, who employs three foresters, works closely with private landowners in two fields, (a) the distribution of free trees each Spring, (b) the preparation of forest management plans.

"500 thousand trees are distributed each Spring in amounts of 1000 to 2500 per landowner after planting sites have been checked. Reforestation is a No. 1 priority in any forestry program projected for Wisconsin. Because of the disastrous forest fires in the past and also because there are millions of acres not suited for agriculture, tree planting is the most effective means of restoring idle acres to productivity. In addition to the hand planting of 500,000 free trees, Trees for Tomorrow also operates a tree planting machine. Through the means of this machine landowners purchase their own trees, furnish the power and labor, while Trees for Tomorrow provides the technical supervision of a forester and prepares the tree planting plan. Last Spring an additional 420,000 trees were planted with a tree planting machine.

"Tree planting is dramatic because in a few years the landowner can receive the results of his efforts. Forest management on the other hand is not dramatic because it requires in many cases ten or fifteen years to see the results of an improvement cut, regeneration by natural growth, or the results from releasing an understory of more valuable trees from an overstory of less valuable species.

"Management of our second growth forest lands is highly important because a good share of our forests today are in a critical growing stage and the wise use of an axe will determine the character and quality of much of our forests of the future.

"The above deals with phases of forestry that can be accomplished as of the immediate present, but I am sure that all of you will agree with me that until we rear a generation of Americans who, first of all, understand what our renewable natural resources are, how they contribute to our social well-being and happiness, how they can be wisely used and renewed, not until then will the security of this nation be assured.

"That is why 50% of the program of Trees for Tomorrow deals with conservation education. Under the heading of conservation education the following projects are being carried out:

1. The awarding of a $1200 forestry scholarship each year to some high school graduate who by his interest and aptitude has indicated he wishes to make forestry a career.
2. The awarding of a $200 4-H Club scholarship to some 4-H Club boy in the state who has shown a leadership in 4-H Club forestry projects. This $200 award is not a forestry scholarship, and it may be used to further his studies in any particular field.
3. The establishment of school and memorial forests that serve as outside laboratories.
4. Operation of the Trees for Tomorrow camp. The camp operates each year from April 1 to November 1. The period from April 1 to June 2 is given over to workshops for high school students. Last Spring during this time, 767 high school boys and girls from 56 schools attended three-day workshops on school time. During the Summer and Fall months 1511 members of adult groups from colleges, members of civic and service organizations attended workshops ranging from three days to one week. Technical meetings are also held for personnel of U. S. Forest Service, the State Conservation Department, industry, and other agencies, while first-year classmen at the Institute of Paper Chemistry spend 3½ weeks each year studying wood technology.

"In summing up progress to date, Trees for Tomorrow has distributed 4,000,000 trees, awarded $12,400 in forestry scholarships, helped establish 34 school forests and 8 memorial forests, prepared forest management plans for 91,000 acres of privately owned woodlands, and has provided workshop programs for more than 8000 people who have attended sessions at the camp at Eagle River. The above is mentioned not from the standpoint that a job has been completed, but to point out how industry can play an important role in forestry and conservation education.

"The job ahead in a region as small as 8 counties, in the entire state, or throughout the country, is a complex undertaking. On any level the work is of prime importance to the social and economic well-being of local communities, to industry, and to the entire nation. It should and can be accomplished at a minimum amount of time by in-the-field action programs and by coordinated efforts toward a single end. This is the voluntary task to which Trees for Tomorrow is dedicated."

HERBERT W. ROWE, *Recorder*
Nekoosa-Edwards Paper Co.
Port Edwards, Wis.

Empire State (Central District)

The principal speaker at the November 9 meeting of the Central District, Empire State Section, was C. E. Foster, Chief Chemist of the Sealright Co., Fulton, N. Y. His talk follows:

Quality Control in the Manufacture of Paper Food Containers

C. E. Foster

TONIGHT I would like to start an argument. In discussing the subject as titled, it is intended to outline in a general way the methods of quality control in a particular conversion operation and then to place some emphasis upon a special problem facing paper converters and the paper producers.

The general purpose of "quality control" is to insure that the product of an operation will meet certain acceptance specifications. Such specifications may be those written by the manufacturer himself based on experience in what his trade will accept as usable, or they may be written by the purchaser to cover his special requirements. A second, and equally vital purpose of quality control, is to help to reduce waste and scrap. Presumably this will be accomplished in two ways: (a) by predicting the production of off-specification goods and, (b) by establishing reasonable standards of quality (practical specifications) preventing the scrapping of usable material and wasting of time in an unnecessary effort to achieve a level of quality in excess of requirements or outside the capability of the process.

The products of our operations are entirely single-service, disposable, sanitary, containers for moist and liquid foods and sanitary closures for glass containers for fluid milk. The nature of such products demands a high productive rate at minimum cost in overhead, labor, and material in order to maintain position in a highly competitive market. One's casual everyday use of such paper products might lead him to think that they are not made to particularly rigid standards, and, generally speaking, in terms of high dimensional precision this is true; however, the demands of the milk and milk products industry in food packaging are high. Consider a milk plant bottling 120,000 units in an 8-hour day (not

C. E. FOSTER, Chief Chemist, Sealright Co., Fulton, N. Y.

unusual): they will apply paper closures at the rate of almost 5 per second. Very much deviation in certain dimensions of these closures, in some cases as little as 0.005 inch, will place the closures on the floor instead of the bottle of milk.

Our quality control operations are divided, broadly, into two phases:

One: Laboratory testing, including raw material, detailed performance tests on finished product, and certain tests and observations requiring a rather high order of technical skill and/or the use of special apparatus.

Two: Line inspection, which includes dimensional specs.; constructional details such as particular grades of paper, color match of printed matter, etc.; such attributes might be termed "operating specifications." Line inspection also is concerned with general quality of workmanship such as proper gluing of joints and appearance of printing; certain performance tests as leakproofness of hot-drink containers; count of units in packages; packaging of product, including protective features such as gluing and taping of cartons and proper identification marking of cartons, or shipping and warehousing.

The laboratory function is to guard over-all quality of product by raw material and process inspection and test. In connection with material specification for new products, the information is given to production by research and engineering departments. Once material specifications have been established, the evaluation of materials from various sources is a joint production-research-quality control function. The evaluation may be a simple laboratory test against certain physical standards such as the boiling range of a solvent, or it may involve a complete test run according to an outline as follows:

I. Check material against physical and chemical specs.
II. Parallel run with material in process.
  A. On production equipment.
    1. Evaluate operating ability.
  B. Pilot performance tests in lab.
    1. Small scale.
      (a) Simulated field conditions.
      (b) Accelerated or destructive tests.
  C. Pilot or small-scale runs followed in actual field performance followed by quality control.
  D. Gradual increase in volume production with continuing field trials followed by sales/service.
  E. Final approval.

The control of individual batches or shipments of material from various vendors is also a laboratory function. Test samples of material properly collated are drawn from each shipment. Physical and chemical tests against specifications are performed in the laboratory. Portions of the samples are actually run through process under supervision of the laboratory and careful observations of performance during process are made. Finished products from the sample runs are then given complete end-use performance tests. In several instances our term "end-use performance" includes functioning in automatic applying and assembly equipment used in the packaging plant. (We actually have in daily use as testing equipment automatic packaging machines valued at from one to fifty thousand dollars.) After the shipment has been approved on a basis of such tests, it is released for use.

In many instances we have as a matter of routine control, arranged with vendors of various materials to furnish us with copies of their own manufacturing test records and in connection with some materials our own personnel act as consultants and inspectors of quality at the vendors' plants. We find it very helpful to have technical representatives of our suppliers spend much time in our plants observing in detail the use of their materials.

Once a lot of material has received laboratory approval, its performance as a lot is rechecked numerous times during the various tests made in connection with process control which is the final laboratory function. Many subassemblies must be checked on a batch basis before they are permitted to pass to the next operation. In some instances such inspection takes the form of a complete trial run as outlined before for raw materials, while in other instances we may only need to make a simple test such as viscosity of an adhesive or coating batch. Every finished product is spot sampled on a representative basis and brought to the laboratory for complete examination as to general appearance and functionability. In the business of producing paper products for the packaging of food, the characteristic of not imparting odor or taste to the contents of the package is of vital importance and especially is this true in regard to milk and milk products such as ice cream, cheese, and butter and certain beverages such as coffee and tea. It is necessary in this connection to train personnel in taste and odor evaluation. While some individuals seem to vary in their ability to detect certain off-flavors or foreign odors, almost anyone can, by training and practice, increase the acuity of his senses of taste and smell so that he becomes a very reliable observer.

L. to r.: C. E. Libby, State University of New York, College of Forestry; J. W. Morrow, Newton Falls Paper Co.; R. N. Prince, American Cyanamid Co.; D. C. Mather, Carthage Paper Co.; K. P. Geohegan, Howard Paper Mills; Henry Perry, Paper Trade Journal; J. S. Reichert, E. I. du Pont de Nemours & Co.; R. D. Moynihan, Finch, Pruyn & Co.; H. D. Cook, Sweet Brothers Paper Co.; R. W. Kumler, American Cyanamid Co.; Barbara Wortley, Solvay Process Co.; J. W. Oren, Armstrong Cork Co.; and F. C. Sommerville, Armstrong Cork Co.

Officers of the Central District, Empire State Section of TAPPI: C. E. Libby, State University of New York College of Forestry, Syracuse, N. Y., Chairman; Barbara Wortley, Solvay Process Co., Syracuse, N. Y., Secretary-Treasurer; and Hilliard Cook, Sweet Brothers Paper Co., Phoenix, N. Y., Vice-Chairman

By training several such people and making tests with panel groups, a fine degree of control may be obtained.

Line inspection operates on a departmental shift basis. It concerns itself with detailed inspection against operating specifications. The procedure followed is approximately as follows:

A. Process inspection.
  1. Set-up check.
    a. Dimensional.
      (1) Gages.
      (2) Capacity—legal tolerances.
    b. Appearance.
      (1) Workmanship.
      (2) Color.
        (a) Match against standards.
    c. Markings.
      (1) Legal requirements.
  2. Running checks.
    a. Continuous during run.
      (1) All above.
        (a) To maintain uniformity.
    b. Performance tests.
      (1) Simple-accelerated.
        (a) Leakage.
        (b) Continuity of coatings where used.
      (2) Interchangeability of parts.
        (a) Control by gages.
        (b) Control by trial—as cover fit and trial on automatic equipment.
  3. Sampling plans.
    a. To secure true representation.
      (1) Must be based upon process variables.
B. Raw material inspection.
  1. Catch spot defects.

In order for a quality control system such as has been described to be effective it is an integrated, active part of the production operation, but in a table of organization it is responsible directly to executives at the managerial level—not production supervision. Line inspectors have the authority to halt production on any unit and to hold up shipment of any questionable product.

There is much interest today in the use of statistical methods of control and some discussion of the application of these techniques may of interest. The use of the control chart on production lines is very limited in extent, partly because so much inspection is by attributes (for example, color matches) and partly because many other defects are completely repetitive when they do occur.

Much value has been obtained from control chart methods in the preparation of batches of material such as adhesives. Once control limits of, for example, viscosity of a given formula have been established, the use of the chart immediately reveals variation in batch properties caused by off-grade material, defective measuring equipment (meters or scales), or operator error.

Statistical analysis of data accumulated in various control operations has been of great value. Some of the information derived includes:

I. Control limits of process are determined to set specifications of performance. In most cases the specification would be for an upper limit only; for example, per cent leakers in a particular type package.

II. Study of effect of process or material changes. When such changes are made to improve product performance statistical analysis answers the question, "Has the product been improved, and by how much?"

When such changes are made for economic reasons, the statistical analysis answers the question, "Has quality been affected and in what manner?"

It is not the purpose of this discussion to delve into the various methods of analysis and their specific applications. Each problem must be handled on its own merits.

In our type of converting operations, once the correct machine setup has been established the process variables which can adversely affect quality are quite readily detected and are usually subject to correction by well-standardized methods. Material variability, on the other hand, while usually easily detected, is not always so readily corrected. This is particularly true of the largest volume raw material: paper.

Of all the raw materials in use in our operations paper shows variation through the widest ranges and in the highest order of frequency. This is a particularly troublesome situation because of all raw materials used it is least subject to correction when once placed in process. Inks and adhesives may be altered in working properties by the use of various additives, and once the alteration is made, the batch will be uniform to a very high degree. About the only way that compensation for paper variables can be effected is to alter other materials or make mechanical changes in equipment and tooling. Often a mechanical change will only be successful in that it enables one to make a choice as to the lesser of two evils rather than correcting the original defect.

It is my considered opinion that of all the materials with which we work, the product of the papermaking process is the least well controlled as to uniformity and uniformity is the heart and soul of any profitable production operation which depends upon low unit profit and large volume for success.

In final conversion operations we take paper which is produced by the ton and reduce it to piece parts which are used by the square inch. If the critical properties of that sheet vary outside of limits in a linear distance of six inches, we are in trouble, either on automatic converting equipment or on automatic packaging equipment using the converted product. The present volume of consumption of paper and paperboard in food packaging is very great and all of us in the paper industry know the potential use is tremendous. What we must recognize is that this potential will only be fulfilled by low cost operation for both the converter and the user of the package. Such low cost will only be obtained through high-speed mechanized operations all along the line and this presupposes a high degree of uniformity in raw material. The paper producing industry has made great strides in increased tonnage per man and machine hour—it must begin to apply its great ability to the problem of uniformity.

Empire State (Metropolitan District)

The Metropolitan District of the Empire State Section met at Fraunces Tavern, New York City, On Tuesday, Nov. 13, 1951. W. R. Willets, Chairman of the District, presided.

K. P. Geohegan, National President of TAPPI, in a short talk expressed his interest and appreciation in being present

Left to right: Ralph Prince, American Cyanamid Co., Secretary-Treasurer, Empire State Section; Kenneth Geohegan, Howard Paper Mills, President, TAPPI; Henry Perry, Paper Trade Journal, Ex-Chairman Empire State Section; Joseph Reichert, E. I. du Pont de Nemours & Co., Chairman, Empire State Section; Ralph Kumler, American Cyanamid Co., National Executive Committee Representative, Empire State Section

and welcomed back the chairman who was prevented from attending the two previous meetings because of illness.

Werner Kaufmann, member of the National TAPPI Executive Committee and long active in TAPPI affairs, recalled that when TAPPI had reached a membership of 1000, many thought almost everyone eligible or interested had joined, but R. G. Macdonald at that time predicted the membership might eventually reach 1500. Dr. Kaufmann said that was one of the very few times Mr. Macdonald had been wrong as the membership was now over 5000.

A panel discussion was held concerning the purchase of paper by large users, with stress on specification buying and its technical aspects. L. S. Reid, Assistant General Purchasing Agent of the Metropolitan Life Insurance Company and past-chairman of the District, acted as moderator and the panel members were R. H. Carter of the Standards Division of the General Electric Co., Schenectady, N. Y.; W. H. Joy of the American Telephone and Telegraph Co.; and M. S. Kantrowitz, Technical Director of the Government Printing office, Washington, D. C.

Following are the talks given:

Purchasing of Paper by the General Electric Co. with Stress on Specification Purchasing and its Technical Aspects

R. H. Carter

The General Electric Co. began purchasing paper to specification in January, 1899, when a specification was issued to cover Black Fiber Tubing for Lamp Shells. Purchasing to specifications has expanded over the years so that we now have some 60 specifications covering approximately 200 grades of electrical papers and 15 specifications covering about 50 types of packaging materials. Practically all papers used in the manufacture and packaging of General Electric products are now covered by specification. Approximately 85% of our annual multi-million dollar purchases of paper and paper products is bought to specification.

The General Electric Co. purchases paper to specifications for three basic reasons: quality, cost, and availability.

The specification tells the supplier just what we want and, therefore, the quality is maintained at the level required for the application.

Since the quality is defined, competitive bids can be obtained and the lowest cost material purchased with full assurance that the material will be satisfactory for the application.

By defining the material, the specification aids in the development of new sources of supply to assure continual availability of material.

Specifications are prepared by the Standards Division for use throughout the entire company. The papers covered are used in such varied products as turbines, motors, transformers, condensers, meters, cables, plastics, and household appliances. With this diversity of end uses, it is obvious that the type of requirements will vary from specification to specification.

We do not attempt to write specifications on all materials purchased by the Company. Rather, we develop specifications only upon receipt of a specific request. In following this policy, we are certain of the need for the specification and have definite assurance that it will be used. The requests may originate in an engineering division, a laboratory, or a purchasing section. Once we receive a request to cover a material, we attempt to write the specification so that it covers all grades of the material used within the Company and in such a way that is suitable for the use of all sections of the Company.

The request for a specification is frequently incomplete insofar as the specific requirements wanted are concerned. It may only mention a trade-named material, a manufacturer's number, or a common name with no indication of the chemical and physical characteristics which may be required for engineering design. What information is furnished is checked against manufacturer's handbooks, existing standards of outside standardizing bodies, and any other recognized standard that may exist so that our specification will cover, as nearly as possible, standard commercially available material. When no data are furnished with the request, the suppliers are contacted for their recommendations.

Only those properties which are needed to assure that the paper will satisfactorily perform its design function are included in the specification. Furthermore, the values specified for these properties are established so as to work as little hardship on the supplier as possible, consistent with the end use. No specification is issued unless at least one supplier agrees to furnish material in strict accordance with the requirements.

Standards have been established for the form and wording of General Electric specifications. In general, each one contains the following elements:

- A scope paragraph describing the material in general terms and indicating the various classes or grades covered.
- Chemical composition of the material, together with restrictions on impurities, moisture content, etc.
- Tabulation of mechanical, electrical, or other properties.
- A section describing manufacturing requirements and restrictions, generally those which cannot be specified numerically.
- Referee methods are listed for all properties required by the specification so that there can be no misunderstanding between the supplier and the purchaser on the manner of measuring these properties.
- Tolerances are listed where applicable.
- A certificate of test clause, which provides for the furnishing of a certificate of test by the supplier, whenever requested by the purchaser.
- Packing and marking requirements to assure that the material will arrive in satisfactory condition and so marked that it may be readily identified.

While I previously stated that only those properties are specified that are actually needed, it is obvious that in a company like General Electric with its diversity of products, one application for a given paper may require that more properties be specified than for another application. The testing of incoming shipments for conformity to the specification is a function of the division using the material, and each division will test only for those properties in which it is interested. The number of tests made by a division for a given property will depend upon how important that property is for the application and how uniform the material may be.

In addition to the specification, which is issued for the use of our purchasing and inspection groups and to inform the suppliers of our needs, we issue related data in two other forms

R. A. Carter, Standards Division, General Electric Co., Schenectady, N. Y

for internal Company use only. The first is for the engineer. It informs him of the composition and property requirements of the specification and provides other available information on the material of a typical nature as well as its uses within the Company. It omits details of the specification he is not interested in, such as referee methods, certificate of test clause, packing, and marking. The second is data for the use of the purchasing and ordering groups. This outlines the proper way to word orders and lists the approved sources of supply for each grade.

An example of the interesting problems of a technical nature that we encounter in specification writing is one involved in the revision of the specification covering the blue bond paper used for all General Electric external stationery. Our present specification includes a requirement that the material shall match as closely as commercially possible the standard General Electric blue color. A spectrophotometric curve is included to indicate the standard color.

We recently ran spectrophotometric curves on several samples taken from different shipments, converted the curves to Munsell color notations, and averaged the results to obtain a new standard color. Then, we plotted tolerance curves around this point for hue, value, and chroma based on what we felt was a good commercial match. Unfortuantely, a majority of the samples fell outside of our tolerance limits.

A supplier arranged all the samples so that there was no noticeable difference between them. Why not broaden the tolerances? When we changed the arrangement of samples, however, a very marked difference showed up. That is exactly our problem. Many more letters are of only one page than of two or more pages; consequently, the stock of second sheets in an office is likely to be much older than the first sheets. Therefore, the second sheet of a letter may not be from the same beater batch, or from the same shipment or even from the same supplier as the first sheet. That is why a close color match is so necessary. We don't have the solution to the problem yet, but we think we are approaching it.

You may be interested to know how we correlate the specification requirements of our various plants. A draft of the proposed specification is prepared, based upon whatever information is furnished to us with the request for the specification, and an incomplete material designation assigned to prevent usage on drawings or orders until it is established as a standard. This draft is referred to the Works from which the request came for approval of their engineers. At the same time, it is referred to the other Works of the Company for their information and for their approval in case they are using the material. Copies are also sent to the various manufacturers of the material for constructive comment and approval. The replies are reviewed and consideration is given to request for additional classes or grades and changes in requirements. In reviewing the replies from the manufacturers, we are generally concerned with whether they can meet the proposed requirements with commercial material and at a cost no greater than for the same nonspecification material. When the comments have been reconciled, the material designation is completed and the specification issued.

Consumer Buying of Commercial Papers on Specification Basis by the Bell System

W. H. Joy

PRACTICALLY all of the commercial papers used by the Bell System are purchased on a specification basis. The use requirements include such varied purposes as records, reports, correspondence, bills, orders, instructions, telephone directories, and miscellaneous publications. The total annual volume exceeds 150,000,000 pounds, or about 4.3 pounds of paper for every telephone in service. Our function at A. T. & T. is the preparation of the specifications and standards. The actual purchasing, inspection, and distributing of the paper is carried out by our affiliate the Western Electric Co. There is, however, close liaison between the different groups.

W. H. Joy, American Telephone and Telegraph Co.

To realize the potential benefits of specification purchasing, it is first necessary to adopt a paper standardization program. This was initiated in the Bell System as far back as 1917. A systematic survey was made of all the many plain papers and thousands of printed forms being used by the different Associated Companies. The results of this investigation showed that in general heavier weights than necessary and far too many colors were being used, and that in many cases the grade selected was either too high a quality or too low a quality for the use requirement, due no doubt to local preferences and a lack of technical knowledge on the part of the personnel involved. Based on this survey it was determined that 13 grades would be adequate and these were substituted for the papers then in use. Particular attention was directed to selecting stocks that would be adequate for the life and service required and yet not more expensive than necessary. At the same time an effort was made to redesign forms of odd sizes to cut economically from mill ream sizes and to restrict colored stocks to a limited number of standards and to use these only for identification purposes. As a result of these measures, coupled with volume purchasing on a direct mill basis, savings in cost on the order of 37% were effected.

The range of stationery form papers has grown over the years from the original 13 to 25 current standards to care for new requirements by the Telephone Companies and technical developments in the paper industry. The types of papers included are ledger, bond, writing, manifold, mimeograph, duplicator, and safety. They vary in quality from 100% rag content for permanent purposes down to groundwood-sulphite Manila stocks for such requirements as temporary use forms and draft copies. Since rag stocks are used primarily for permanence, only papers of 50% rag content or higher have been standardized on the theory that at least half the fibers present should be of a permanent nature. On a poundage basis, only about 10% of the total annual volume are rag grades. Names of our own devising have been established to cover the various grades, such as Telco Cable Ledger, Bell Seal Bond, Telcopaque Bond, etc. The Operating Companies are provided with a paper chart which lists the various standards, their characteristics, and recommended use requirements. The fieldman uses this chart to select a suitable quality for the form in question.

Our paper specifications were a natural outgrowth of the standardization program. With the introduction of volume purchasing a laboratory was established to check the quality of the deliveries. It early became evident that some yardstick was required to determine whether or not the sample under examination was of satisfactory quality. Inspection limits were accordingly established based on accumulated test data and actual field experience. From these somewhat crude beginnings we now have in effect 62 technical specifications covering paper and paper products. Most of the specifications include the following requirements: weight, thickness, bursting and tearing strengths, ash, pH in the case of permanent grades, brightness, opacity, and fiber content. On cut sheets long grain direction is specified. We have found that for the most part we can depend on mullen and tear for physical strength so that tensile strength and folding endurance are only used in a limited number of cases. The requirements are on a maximum or minimum basis; in some cases, for example, weight and thickness, both are given. Where the grain direction affects the test, as in tear, the same minimum is given for both directions rather than an average value on the theory that a paper is only as strong as its weakest direction. All specifications are supplemented by approved samples to cover such features as shade, dirt, finish, formation, sizing, writing and erasing, and printing and duplicating characteristics.

In preparing specifications effort is directed toward describing the commercial grades required in terms of fully practical and reasonable quality limits. Any existing specifications or trade customs are taken into consideration and where possible limits having general recognition are adopted. Frequently tentative specifications are submitted to a number of representative mills making the type of paper in question and the final requirements adopted are a result of this cooperative effort. In establishing the minimum strength requirements a margin of safety is allowed to care for the unavoidable variations during a mill run.

It is expected that all of the paper delivered will conform with the specifications. This, however, does not always happen. Good judgment should be exercised in the interpretation of delivery inspection tests. When nonconformities are found, we have a choice between three courses of action. If the deficiency is of a minor character, it is simply ignored. If more serious, but still not sufficiently so as to affect the utility of the paper, it is brought to the mill's attention for correction on the next mill run. In rare cases where a flagrant deficiency is found that would seriously affect the use requirement, the paper is rejected. For the most part, inspection tests are made on mill outturns to check the quality of the paper before it is shipped. On directory paper involving large tonnage, the Western Electric Co. has inspectors stationed at the paper mills to check performance on the spot so that deficiencies can be immediately rectified. Several thousand paper samples are tested annually in connection with the quality control of our papers.

Specifications should be flexible and subject to revision with changing circumstances and technological developments. On the other hand, we do not believe in lowering specification requirements to care for temporary conditions. For example suppose the mill is having difficulty meeting a minimum bursting strength limit of 33 points due to unavailable raw materials, or possibly because they are running at faster speeds than normal to step up production. Actual tests show mullen data around 30 to 31 points, and no field difficulties are being experienced. If the minimum is dropped to 30, it will not be long before the paper will be running in the 27 to 28 range. It seems to be human nature for the mill superintendent to shoot at the new rather than the old limit with a gradual depreciation in quality. A similar situation currently exists in connection with brightness. Where the condition is beyond the mill's control and expected to last for an indefinite length of time, temporary amendments are issued to supplement the specification proper.

In conclusion experience over many years has indicated that the Bell System has obtained the following advantages from paper standardization and specification purchasing:

1. Standardization eliminates duplication of effort. One central agency rather than each Telephone Company is concerned with the paper problem. Proper grades are assured for each use requirement.
2. Savings are achieved due to volume purchasing on a tonnage rather than a poundage basis. Specifications permit competitive bidding and are helpful in developing new sources of supply. The mill has a good idea before running the paper what is desired.
3. Printing contracts are negotiated on a square inch basis. Since the paper is furnished at a set cost, in formulating his bid, the printer is concerned only with those elements of cost over which he exercises direct control.
4. Quality control is assured. Long runs of the same grade and weight enable the mills to more readily maintain uniformity. Routine inspection procedures have a salutary effect in keeping deliveries up to specification minima.
5. Weight control results in direct dollar and cents savings. On roll paper which is purchased on a weight basis and used on a yardage basis; such as directory paper, certain toll ticket papers, and addressing machine bill paper; overweight will result in fewer books or forms from a given quantity of paper. Overweight can also adversely affect mailing costs on publications such as our annual report which involves around a million copies.
6. Field difficulties and compaints are kept to a minimum since the paper is checked prior to shipment. When disputes do arise the specifications provide a basis of discussion with the mill, the printer, or the ultimate consumer in the field. Quality is not simply a matter of opinion. A definite yardstick is provided for evaluating it.
7. Due to our large volume and reasonably constant demand, long-term contracts can be made with reliable mills. During times of stress, such as the present paper shortage, these assured sources of supply have proved their worth.

Technical Aspects of Government Printing Office Paper Specifications

Morris S. Kantrowitz

THERE is no commercial enterprise more closely related and more dependent upon papermakers than the printing industry. The Government Printing Office was established by authority of Congress in 1861 to execute printing and binding for the Senate and House of Representatives and the Executive and Judicial Departments and for the Court of Claims. Today the U. S. Government Printing Office constitutes the largest printing plant in the world and its functions extend to all branches of the Government. During the last fiscal year, which ended June 30, 1951, it produced $98,222,845 worth of printed matter.

The Government Printing Office purchases all classes of paper from newsprint to the highest quality ledger and index paper on technical specifications. Last year, purchases were made for 186,990,300 pounds of paper and 81,085,700 envelopes.

The paper purchases for this period amounted to $27,390,-206 and for envelopes $308,865. The Schedule of Paper for the year specifies 82 grades of paper under 174 items furnished by approximately 70 different mills.

I should like to indicate to you the quantities of some of the more important grades of paper purchased last year.

Grade of paper	*Pounds*	
Newsprint	7,500,000	
Coated book	14,439,000	
Uncoated book	36,684,000	
Mimeograph:		
Rag content	1,981,000	6,199,000
Chemical wood	4,218,000	
Map	7,198,477	
Writing	43,079,000	
Bond	13,035,000	
Ledger	5,144,000	
Index	6,880,000	
Postal card	25,167,000	

One of the largest single items is for 25,167,000 pounds of postal card paper. The contractor for this item must deliver approximately 80,000 pounds daily to keep pace with the production of United States postal cards at a rate of 384,000 per hour on each of four presses.

The law permits for contract periods of three, six, and twelve months and, in recent years, these contracts have been established for three-month periods. In addition to regular contracts, the Office makes hundreds of purchases in the open market. These purchases consist of Government forms, tags, various envelopes, jackets, and other paper and paper products not provided for in the schedule for contracts.

Government paper specifications are formulated by two different committees. Those for papers purchased by the Government Printing Office are formulated by a committee appointed by the Joint Committee on Printing of Congress, while the specifications for papers not covered by the Government Printing Office are formulated by the Paper Technical Committee of the Federal Specifications Board.

M. S KANTROWITZ, Technical Director, Government Printing Office, Washington, D. C.

The Committee on Paper Specifications of the Joint Committee on Printing is composed of the Clerk of the Joint Committee on Printing, who serves in the capacity of Chairman; an Inspector of the Post Office Department; the Inspector of Paper and Material for Joint Committee on Printing; the Chief of the Paper Section of the National Bureau of Standards; and three Officials of the U. S. Government Printing Office, namely, the Technical Director, the Director of Purchases, and the Chief Storekeeper.

New developments in paper manufacture, and in the printing and binding industries, and new requirements of all Governmental departments are considered periodically by this Committee as required, in the formulation of specifications before each contract period. As a result, a specification of the U. S. Government Printing Office for any grade of paper represents the latest development in that particular grade.

The Paper Technical Committee is appointed by the Federal Specifications Board. It is composed of the Chief of the Paper Section of the National Bureau of Standards as Chairman, the Technical Director of the Government Printing Office as Vice-Chairman, and representatives chosen from the interested Governmental executive departments.

This committee prepares specifications for the purchase by various Government departments of such papers as blueprint, carbon paper, cross section and drawing papers, teletype, and many others for use in the field services and in the departmental services where Government Printing Office specifications do not exist.

The use of technical specifications is not only beneficial to the Government, but is equally beneficial to the paper industry as a whole. It assures the buyer of equality in the kinds of paper offered by competitive bidders, and also assures the manufacturer or supplier that all competitive bids are based upon identical qualities in the paper. The paper specifications define the minimum quality of paper required for particular uses.

All deliveries of paper and envelopes for public printing and binding are tested by the Government Printing Office Laboratory for compliance with these specifications. The testing consists of two parts: first, technical testing covering the microscopical, physical, chemical, and printing properties; and second, visual examination of the paper for comparison with standard samples.

The Government Printing Office each year issues a sample book containing a compilation of standard samples of the various papers proposed for purchase. Unbound standard samples are furnished to bidders and contractors for their guidance as to the requirements for color, formation, and cleanliness, which cannot be readily specified in numerical values.

Fiber Content. The fiber content of practically all papers purchased by the Government Printing Office is specified under the requirement designated as "stock." It gives some information as to what may be expected in permanence and use of the paper.

Beginning with March 1, 1929, bond and ledger papers for the Government Printing Office have been watermarked to show the rag content. The watermark consists of the seal of the United States with a halo of one star for 25% rag paper, two stars for 50%, three stars for 75%, and four stars for 100%. The chemical wood papers are not watermarked.

Weight. Weight is specified on all purchases of paper for the basis of cost.

Physical Characteristics for Evaluating Durability. The specifications covering the physical characteristics include bursting strength, folding endurance, tensile strength, and tearing resistance to insure durability.

Bursting Strength. The bursting strength test is one of the oldest and most widely used, particularly as a control test by paper mills for measuring the strength of practically all grades of paper and it is thus included in the Government specifications. The results of the test give an indication of the ability of a paper to stand up under a bursting strain.

Folding Endurance. Folding endurance is an index of the resistance of paper to repeated folding. This test is generally regarded as the most valuable for papers such as writing, bond, ledger, index, and cover, which are required to undergo considerable creasing and folding in use.

Tensile Strength. The tensile strength is indicative of the serviceability of many papers which are subject to direct tensile stresses. In the case of printing papers, tensile strength measurements indicate the potential resistance of paper to breaking when subjected to strains such as during its travel from the roll through the web press mechanism in the process of printing. It fills a special need in the specifications for paper such as that used in printing newspapers, magazines, and textbooks on web presses.

Tearing Strength. The Government Printing Office specifications include a tear test on papers which are subject to tearing strains in actual use, such as wrapping and high wet-strength map papers and tag stock.

Chemical properties of paper are included in the specifications for the purpose of procuring papers of maximum permanence. These include acidity, rosin sizing, alpha-cellulose content, and copper number.

Acidity. A very accurate analysis for acidity, or determination of the pH value of paper, is most important in the case of rag content book, bond, ledger, and index papers intended for use in permanent records. Paper intended for permanent record use should be required to have a pH value of not less than 5.0 when determined by hot extraction.

Excessive Rosin Sizing. Rosin is detrimental to the life of paper when present in excessive amounts, since it tends to produce discoloration and deterioration of the paper constituents under the action of light. It, therefore, becomes necessary to write this qualification into the specifications of certain types of papers, and careful analysis is made to determine the quantity of rosin present.

Alpha-Cellulose Content. Some believe that chemical stability constituting permanence of paper is related to the alpha-cellulose content of a pulp, contending that the higher this content is, the more permanent the paper. Others state that, in some instances, considerable fiber deterioration may occur before there is an appreciable loss in the alpha-cellulose content. Still others state that this test is significant only when comparing the alpha-cellulose content of fibers of the same kind and origin.

In Government specifications, a requirement of not less than 95% alpha-cellulose is included in the 100% rag content book, bond, ledger, and index papers intended for permanent record use.

Copper Number. The copper number is regarded as a direct index of those impurities in cellulose such as oxycellulose, lignin, and sugars which possess reducing properties. It is sensitive in detecting changes accompanying deterioration and may, therefore, be considered a good test for evaluating the permanence of paper.

The copper number of all permanent record book, bond, ledger, and index papers purchased by the Government must not exceed 1.0.

In the group of paper characteristics included in G.P.O. specifications which are most important from a printing standpoint are smoothness, ink receptivity, surface bonding strength (pick test), opacity, thickness, moisture content, formation, color, and general appearance.

Smoothness. Smoothness may be regarded as an index of the facility of a paper to make close contact with a printing surface under the influence of pressure. For the purpose of letterpress printing, smoothness is therefore one of the most important printing properties demanded of paper.

In Government specifications, the smoothness of printing papers refers to their finish.

Ink Receptivity. The penetration of printing inks into absorbent paper, such as news and book, is dependent primarily upon the vehicle used in the manufacture of the ink.

An oil penetration requirement included for book and mimeograph papers and tests for conformity to the values specified are a help in selecting the proper paper and ink and indicating their performance on the press.

Surface Bonding Strength. Surface bonding strength or resistance to picking is another important printing quality demanded of paper, especially in coated stocks and in uncoated papers used in printing halftones by the offset process of printing where tacky inks are used.

Picking leaves small white spots on the printed image and is especially noticeable in solid areas. While picking may be reduced by the use of compounds in the inks, which reduce their tack and their pulling tendency, it results in a gray or faded appearance of the print. The most desirable solution of this problem is to make the paper surface resistant to picking influences.

Opacity. Printing demands paper having a high degree of opacity, especially when halftones or solids are printed on one side of the sheet with type or light printing on the other side. Deficiency in opacity is responsible for show-through of printing which interferes with legibility and also causes a bad general appearance of the work.

In Government specifications, opacity is expressed in terms of contrast ratio.

Thickness. Uniform paper thickness is essential for good printing. Variation in thickness of paper results in variations in blackness or color intensity of the inks and interferes with the consistent printing quality of the sheet.

Thickness of book paper is of paramount importance in controlling the bulk of the finished book. Individual volumes of a set of books printed on the same grade and weight of paper and containing the same number of pages are required to have the same bulk.

Index paper must not exceed the specified thickness in order to economize filing space.

Thickness of U. S. postal card paper is important in relation to the printing and production operations.

Moisture Content. The measurement and control of the moisture content of paper, particularly that intended for multicolor work, is essential in the production of quality printing. Moisture content should be specified for the control of register, static, cockling, creasing, and curling.

Paper should be ordered from the mill with a slightly higher moisture content than that of the pressroom air. Lithographers with no special paper conditioning equipment should specify a moisture content of 0.5 to 1.0% above equilibrium of the usual pressroom humidity for their printing papers.

Color. In paper specifications, color is generally referred to simply as "color" such as white, green, blue, pink, buff, and must match a selected standard.

Variation in color of book paper, writing, bond, and ledger papers is not only encountered among shipments from different contractors, but sometimes occurs within the same shipment. The difficulties which arise from variation in color of paper, cannot be corrected in the pressroom. On a long-run job, it is sometimes necessary to supply the pressroom with paper taken from different shipments. If the color of successive lots of paper differs during the press run and the ink remains the same, there will be a variation in the tone value of the printed page, since the ink formula and its tonal quality have previously been adjusted to the color of the paper first used.

Formation. Formation has been defined as the uniformity with which the paper fibers and other solid components of the sheet are distributed. It concerns the appearance of the sheet when viewed by transmitted light. This property is very important not only because of its influence on the appearance of the sheet, but because it influences the values and uniformity of values of nearly all other properties. A closed and uniform formation is an essential item in good printing papers. Without this, regardless of other paper qualities, only inferior printing can be done. When this is the case the ink will be unevenly transferred and unevenly absorbed, resulting in a mottled and spotty appearance of the printing.

General Appearance. One of the most objectionable features of paper is an unsatisfactory general appearance due to the inclusion of dirt. The term "dirt" may be applied to any foreign material embedded in the sheet such as black mineral and resinous specks, bark specks, shives, pieces of wood, rubber, wool hairs, color spots from undissolved dyes, oil, grease, and wax spots. The presence of even minute particles of dirt, scarcely discernible by the eye, affects the utility of the paper. It destroys the effectiveness of good printing and this is particularly degrading to the highlight effects in halftones. Gritty particles of dirt wear down the copper printing plates and halftones. In rotogravure printing, they scratch the polished nonprinting surface of the copper cylinder, causing fine hairlines to be printed in the nonprinting areas.

Dirt in fine writing, bond, ledger, and index papers produces an unsatisfactory appearance which affects their sales.

In concluding, permit me to say that we in the Government Printing Office appreciate the excellent cooperation which you, the technical men of the paper and other related graphic arts industries, have always so freely given us.

The talks gave rise to an interesting discussion period. Some of the questions raised, together with the answers follow:

Q.: Is there any method for measuring formation?

A.: There is no method for numerically defining formation, but the quality is determined by a visual inspection.

Q.: When a government specification designates a particular type instrument, such as the Bekk Smoothness Tester, can a mill that does not have this instrument substitute results obtained with a different type tester?

A.: It is up to the mill to obtain the correct type instrument, and specifications must be met according to the standards issued. However, the mill may be able to correlate its instrument with the type designated in Government specifications by submitting various samples which will be tested on the particular instrument n question. These results may then be compared with those obtained by the mill, and a correlation reached. This correlation of the two instruments will only be accurate for the same type paper.

Q.: In giving the specification on color for the blue paper used by General Electric, the standards seem to be set up based on the G.E. Recording Spectrophotometer. Since many mills do not have an instrument of this type, how are they to meet specifications?

A.: General Electric plans to set up a series of blue standard sheets, based on the Spectrophotometer curves and these sheets will be distributed to the various suppliers. It will then be up to the mill to match the color by visual comparison with the standard sheets.

Q.: Is any specification for two sideness usually given?

A.: No numerical specification for two sideness is usually given, but it must meet a visual test.

Q.: Does the printing ink absorption of uncoated papers vary very much?

A.: The ink absorption will be markedly affected by the type and amount of filler used. With a given filler the ink absorption will also be affected by the amount of hydration obtained in the beating operation. The smoothness of the final sheet will also affect ink absorption.

Q.: In some specifications for Government letterhead paper, no specification for opacity is given, why is this?

A.: It is felt that since most letterhead paper is written on only one side, show-through is not important and no specification on opacity is needed.

Q.: It was mentioned that the brightness specifications on many papers may have to be lowered, what is the reason for this?

A.: Because of the chlorine shortage, mills may be unable to meet brightness specifications.

L. E. Georgevits, *Recorder*
Titanium Pigment Corp., New York, N. Y.

Empire State (Eastern District)

The Eastern District of the Empire State TAPPI held a meeting on Nov. 15, 1951, at which 50 members heard an interesting paper given by Joseph F. Baigas, Jr., Engineer for the National Council for Stream Improvement, Inc., New York, N. Y.

Ralph Prince, Secretary of the Empire State Section, was present and told about the junior prize contest.

Mr. Baigas was introduced by E. H. Johnson, our Program Chairman. His paper follows:

The New York State Pollution Control Law of 1949 in Action

Joseph F. Baigas, Jr.

Previous to the enactment of Chapter 666 of the laws of the State of New York, the control of pollution was vested in provisions of the laws of the State including the common law and various enacted statutes. These laws were thought not specific enough to control stream pollution and protect the public interest, so after much deliberation, the Water Pollution Control Law of 1949 was enacted. It is very interesting to note that this act was passed in the legislature without a single dissenting vote.

The law has the following important provisions:

1. The creation of a Water Pollution Control Board empowered to abate and prevent pollution in accordance with the provisions of the law.
2. The classification of the waters of the State and the adoption of standards of water quality thereof.
3. With respect to existing discharges, any person who, at time of enactment of the law, was discharging sewage or industrial waste shall discontinue such discharge if it causes pollution as defined by the standards of quality to be established. If no known reasonable method of treatment is available a period of 5 years is alloted for development of such methods. This period may be extended, if necessary, after public hearing.
4. After the date of the law any person who wishes to make any new outlet for the discharge of waste shall first apply to the board for a permit to do so.

If we analyze these provisions, it can be readily seen that the most important provision in enactment of this law is the classification of waters and adoption of standards of quality thereof. Prior to these classifications, the board cannot act to reduce waste discharges except in emergency cases where the public health is endangered. This might be stated in another way. That is, according to the definition as implied by the law, there is no pollution existing in any specific waters of the State until classification and the application of standards are complete for these waters. After classification any waste that shall contribute to a controvention of these standards shall be considered unlawful and subject to the abatement provisions of the law.

In recognition of the importance of this classification system the law has provided that the classifications be reasonable and based on the best uses which have been made, are being made, or may be made of the waters. Seven possible classifications have been established:

1. AA, drinking, culinary, or food processing after disinfection.
2. A, drinking, culinary, or food processing after coagulation, sedimentation, filtration, and disinfection.
3. B, bathing.
4. C, fishing.
5. D, agricultural and industrial supply.
6. E, sewage and industrial waste disposal and transportation.
7. F, sewage and industrial waste or other waste disposal only.

For each of the afore-mentioned classifications, certain minimum standards have been established. Those items affecting the pulp and paper industry relative to their discharge are those which refer to:

1. Floating solids, settleable solids, oil, sludge deposits, taste, and odor-producing substances.
2. pH.
3. Dissolved oxygen.
4. Toxic substances, deleterious substances, and colored wastes.

In line with their powers to abate pollution and the definite procedure established by law in so doing, the Water Pollution Control Board has followed a comprehensive plan of action which consists of, first, the investigation or sanitary survey of the various drainage basins throughout the State, and, second, the publication and distribution of a report on the result of these surveys. A public hearing is then held at a convenient location within this drainage area at which all interested parties may voice their opinion on the classifications. Finally, the waters are classified and the appropriate standards of quality assigned. After official classification, a plan for the abatement of pollution as defined by the classifications must be prepared by the Board. This includes a time schedule of what the Board considers reasonable time for the preparation of plans, commencement of construction, and completion of works. A conference is then held with violators to see how far they will go in proceeding voluntarily to correct conditions and finally the program is put into effect. Those not complying with the orders of the Board will then be subject to the enforcement provision of the law.

The State program has operated since 1949 consistent with this procedure. It was originally estimated that the classification of all the waters in the State would be complete by 1959. However, unless additional staff is provided, the executive secretary of the Board, A. F. Dappert, states that it will be 1964 or 1965 before the classification of all waters is complete. Since classification of waters is necessary before any abatement program can be enforced, the survey and classification program has been concentrated on basins where real acute pollution problems exist. Such a policy will continue until additional help is acquired.

According to data received from Mr. Dappert, field surveys on watersheds representing about 7% of the total area of the State have been completed as of September of this year. These include the following basins:

1. Roundout-Wallkill (900 sq. mi.).
2. Moriches Bay (70 sq. mi.).

Hearings were held in these two basins this year and the waters have been officially classified. Work is now in progress on the comprehensive programs for the abatement of existing pollution as defined by the classifications.

3. Conewango-French-Brokenstraw (1000 sq. mi.).

Survey report on this basis is now being printed and the report will be published shortly.

4. Onondaga Lake (400 sq. mi.).

Survey report being printed and will be published in a few months.

5. Sauquoit Creek (70 sq. mi.).

Report to be published shortly.

6. Mill Creek (30 sq. mi.).

This report has been published and hearing will be held on November 20.

7. Big Sister Creek (50 sq. mi.).

Report has been published and the hearing was held on October 24. The official classifications will follow shortly.

8. Olean Creek (180 sq. mi.).
9. Peconic Bay (100 sq. mi.).
10. Esepus Creek (150 sq. mi.).
11. Sawmill River (45 sq. mi.).
12. Sparkehill Creek (30 sq. mi.).

On these five small basins the surveys have been completed and the reports in various stages of completion. Survey work is now under way on the Ramapo River and should have been completed last month. Preliminary work has been completed on the Delaware River Basin (2500 sq. mi.), but completion of the work is 2 to 3 years off. Some studies are now in progress on the lower Hudson in cooperation with

Joseph H. Baigas, Jr., Engineer, National Council for Stream Improvement, Inc., New York, N. Y

the United States Fish and Wildlife Service in connection with the shad problem and survey work is under way on a portion of the Mohawk Basin exclusive of the subwatershed of Schoharie and the two Canada Creeks.

After completion of the classification programs on the various basins, the work of the Water Pollution Control Section is extensive. Under the present organization, the lack of personnel and funds is jeopardizing the efficiency of the program. Mr. Dappert, executive secretary of the Pollution Control Board, stated to a recent meeting of the Isaac Walton league: "We are reaching the stage now that we absolutely must have more help or the program will begin to bog down. According to the law, once we have classified the waters, we must develop a comprehensive plan or program for proceeding to abate the pollution. All of this will require a great deal of detailed work. To enable us to carry forward the present survey classification program at a somewhat faster rate, but particularly to enable us to carry forward the development of comprehensive programs as rapidly as the waters of the various basins have been classified we have requested additional appropriations. We shall keep on asking for and presenting justifications for staff and facilities somewhat commensurate with our actual needs."

Although it might be advisable to review in detail the classification of the Roundout-Wallkill basin and Moriches Bay at this time, these are so detailed that it could not possibly be done in one sitting. In the Roundout-Wallkill River Basin which comprises a 900-sq. mi. drainage area, there are 391 separate classifications. This gives some indication of the tremendous job confronting the State authorities. All possible classifications were utilized in this work except Class E. The Roundout River from mouth to Eddyville was classified Class C and from Eddyville to the Wallkill Class B. The Wallkill River from Roundout River to Rio Grande received a B classification and from Rio Grande to the State line Class C. The various tributaries received classifications ranging from AA to F excluding E. A "D" classification was given to most of the small pasture streams serving primarily agricultural and drainage purposes. There are several sections of tributary streams classified Class F and to quote Mr. Dappert's remarks to the Isaac Walton League regarding these classifications: "You may wonder why we should consider any classifications as low as F. I shall give you an example. A section of Monhagen Brook below Middletown has been officially classified as F. Were it not for the sewage flow from Middletown, Monhagen Brook would be essentially a dry stream. It is absolutely imperative that the City of Middletown utilize this stream as a means of sewage disposal. The only way in which this stream below the city could ever be restored to something resembling its pristine purity would be to move the City of Middletown off the watershed. Being realistic, I think you would agree that this is an impossibility. Therefore, the best usage for Monhagen Brook immediately below Middletown has been judged to be for sewage disposal purposes and accordingly placed in Class F. Now this classification by no means permits the City to dump untreated or inadequately treated sewage into the brook. We can and do expect the City to improve its sewage treatment facilities to the point that the stream will no longer be the public nuisance that it has been for many years. As a matter of fact to meet the standards for Class F the City of Middletown is required to install treatment works providing for a very high degree of treatment which it is now doing."

This statement by Mr. Dappert is important to the industry for in instances where a similar situation might occur with regard to pulp or paper mill wastes, we should expect similar application of the F classification for since it is unreasonable to remove the City of Middletown from the drainage area, it likewise would be unreasonable to move a pulp or paper mill from the drainage area. In general it is believed that the classifications are very reasonable and in accordance with best usage. As a predator of classifications to come, particularly those which will affect the paper industry to a greater degree, these indicate a very thorough and sound approach by the Water Pollution Control Board in the establishment of the basis upon which the success of the Pollution Control Law of 1949 is dependent.

Concurrent with the State's program of stream classification, the paper industry, under the New York section of the National Council for Stream Improvement, has been conducting water quality surveys in the areas of particular interest to the industry. The purpose of this work is to determine the intensity of any problem that may exist and also since the law affords all interested parties a definite voice in deciding classifications, to gain factual data so that the industry can enter the classification hearing and discussions on a sound basis. The general procedure followed in these surveys is for the engineers of the National Council to plan and lay out the details of the survey and then, with the assistance of personnel of the mills along the particular river, carry out the sampling program and conduct the necessary analyses. On completion of the field work, a report is written wherein the existing sanitary conditions of the stream are defined and the possible classification of the waters estimated with reference to these conditions and the existing and possible future uses of these waters. To date, five surveys have been completed. Last year, the Black River including the Beaver and Moose tributaries, Sauquoit Creek, and the upper Hudson River from Corinth to Thomson were surveyed and this past Summer a survey of the Lake George Outlet and the Oswego River were completed. Operations this Summer were partially hampered by general high flow conditions due to the abnormal rainfall during July and early August.

It can generally be stated that the sanitary conditions of the waters receiving pulp and paper mill wastes in the abovementioned drainage areas are good. Indications are such that, if the classifications of these waters are reasonable and follow present usage as were those of the Roundout-Wallkill basin, only minor abatement measures will be required by very few of the mills.

I have previously mentioned several specifications of the classification standards that might affect the paper industry. I would like, at this time, to summarize generally the average results with respect to these items.

1. Floating solids; oil; sludge deposits.
   The standards for Class B and C, those which most of the waters of the completed surveys will meet, are alike—none which are readily visible and which measurably increase amounts . . . after mixture with the receiving waters.
   (a) Floating solids.
   The floating solids noted were foam, bark, and wood chips from pulping operations, and sludge islands caused by the decomposition, gasification and rising of fiber deposits.
   (b) Settleable solids.
   Settleable solids were noted not so much because there were measurable increases below mill outfalls after mixing, but that they are in some instances, readily visible.
   (c) Sludge deposits.
   The sludge deposits traceable to paper mill wastes were not extensive, but in some cases might contravene the standards. These deposits originate mostly from white water solids.
   (d) Oil.
   Oil is a minor item and although some oil was noted below mill outfalls improved housekeeping should reduce oil discharge to a point where it is no longer visible.
2. pH.
   pH is not a problem. In all watersheds surveyed the average pH was within the required range, 6.5 to 8.5.
3. Dissolved oxygen.
   Standards for this item dictate a minimum of 4.0 p.p.m. for nontrout waters and 5.0 p.p.m. for trout waters. Average dissolved oxygen in all waters surveyed was above 4.0 p.p.m. and there is generally no indication of an overload of the purification capacities of any stream.

4. Toxic wastes, deleterious substances, colored or other wastes.

No problems of any extent are caused by waste from the paper industry having these properties except some local color problems. Color is a poor indicator of stream pollution from the viewpoint of dissolved oxygen, pH, and suspended solids for we can have excellent condition with regard to these items but still have residual color. In addition, color is one of the layman's pollution yardsticks for he usually measures pollution by what he sees or smells. This color item, where important, will have to be resolved at the hearings for removing color particularly from pulping waste is a difficult problem. An example of what may happen with respect to color may be something like this. Assume a certain water will meet Class C standards for all items but there is a residual color from a mill. Now as far as science knows color alone is not detrimental to fish life but it can be seen that color will affect the fisherman. What might be asked is the C classification to protect the fish or to protect the fisherman. You can see how such a situation might develop resulting in pressure on the industry to remove the color. Therefore when a color problem exists or might exist due to pulp or paper mill wastes, the industry must take an active part in the hearings so that an unreasonable burden is not forced upon them.

The survey program of the New York section of the National Council for Stream Improvement will continue next Summer. Surveys have been requested on a portion of the Mohawk River and the Hoosick River. Following this work, the staff of the Council will be available for other surveys where and when requested.

The question period after the paper included the following discussion:

Question: What is the status of a program for abatement on surveys already made?

Mr. Baigas: There will be no enforcement until the survey is made on all watersheds. Until the survey is made, it would not be fair to one area to clean up when another area may be a worse offender. The surveys cannot be finished before 1963 unless additional personnel and funds are made available to the Council.

Peter Abel: Is 10,000 gal./ton effluent a good figure for a board mill with recirculation and should a mill have equipment to clarify that amount?

Mr. Baigas: 10,000 gal./ton is a good figure for the effluent. Before installing equipment to clarify this, a mill should recirculate as much as possible.

R. Prince: You mentioned one area the State and the Council have surveyed. Has there been any estimate of the cost to make the necessary improvements?

Mr. Baigas: No, as the advisory board that recommends the improvements is not operating as yet.

Question: What has resulted in other states that have had to clean up? Does the stock saving pay for an installation?

Mr. Baigas: White water clarification will bring returns, however, material from stock washers, cooking liquor, or material that colors the water will not.

Question: Do you have anything on the waste sulphite liquor problem?

Mr. Baigas: This is an economic problem. A base change may clear up the problem but would be costly in view of the capital investment already made. Research projects are going on to seek an economic treatment.

Question: Are the results of individual mills research being made to the National Council?

Mr. Baigas: Most of them are being made known, such as the OCO system that returns crystal clear water. The National Council is conducting research projects at Rutgers, Virginia Polytechnic Institute, Louisiana State University, Kalamazoo College, Institute of Paper Chemistry, Mellon Institute, and Oregon State College. It is interesting to note that 90% of the paper industry are members of the National Council.

H. H. Lavery thanked Mr. Baigas for his interesting presentation. The next meeting will be Dec. 13, 1951.

R. D. Lewis, *Publicity Chairman*
Eastern District, Empire State Section

Pacific

On Nov. 13, 1951, approximately 200 men attended the annual engineering meeting sponsored by Pacific Section. This year's session was held in the spacious auditorium of the Lower Columbia Junior College at Longview, Wash. Donald G. Felthous, Pulp Division, Weyerhaeuser Timber Co. Longview, Wash., served as moderator at the afternoon technical meeting. The speakers were L. E. Hill, Jr., Weyerhaeuser Timber Co., Everett; Harold Springer, Rayonier, Inc., Port Angeles; James E. Maxwell, U. S. Department of Commerce; Donald M. Platt, Crown Zellerbach Corp., Camas. Arthur E. Erickson, Pulp Division, Weyerhaeuser Timber Co., Longview, was in charge of arrangements for the meeting.

A reception for the group was held at the Hotel Monticello at the conclusion of the technical meeting, followed by dinner.

Walter J. DeLong, Division of Public Information, Weyerhaeuser Timber Co., Tacoma, addressed the dinner meeting on the economic and political problems confronting the nation.

The next meeting of Pacific Section will be on January 15 at Olympia, Wash., at which time the subject of Process Control will be discussed.

A résumé of the technical papers follows:

1. Observation on Chip Handling from Barges and Cars, by L. E. Hill, Jr., Plant Engineer, Pulp Division, Weyerhaeuser Timber Co., Everett, Wash.

Production of chips at sawmills is becoming a standard operation, requiring pulp mills to install unloading systems best adapted to type of carrier, mill flow pattern, and adequate to their competitive share of this chip market.

Chips present a difficult unloading problem because chips consolidate and interlock in a bulk carrier. This occurs whether carried by ship hull, box-top scow, railroad cars, or automobile truck body.

Gravity unloading from bottom dumping rail cars requires rodding or vibration to obtain flow. Gravity unloading from trucks can be obtained by opening tail gate and raising body to a steep angle. Roll-over unloading of rail cars requires a heavy investment and is only justified by a large number of incoming cars.

Hog fuel type of clamshell bucket is being used, but presents the problem of effective clean-up of scow. Smaller equipment of similar type has been used to unload rail cars but was slow and damaged the cars.

Pneumatic suction is used at several mills because it unloads and transports with one unit. Can be used in ship hold obstructed by beams or in standard box car. Systems normally require hand labor to keep suction pipe supplied with chips. Consumption of power is an important item in costs.

Cars fitted with cable-drawn plows in their bottoms are being unloaded into pits. Chips come out of side doors or bottom doors, but must be manually freed from side walls before plow can be used.

Ordinary box cars or open top cars could be unloaded with a scoop truck or by hand, but rate of unloading would be too low to fit into flow pattern required by most mills.

At present there is no one system which best fits all situations, therefore selection of a system must follow from consideration of all variables at the individual mill.

2. Protection of Electrical Equipment Against Corrosion in Industrial Plants, by Harold E. Springer, Chief Electrician, Rayonier, Inc., Port Angeles, Wash.

Corrosion of vital parts of electrical equipment presents a serious problem in many industries where corrosive gases, dusts, or liquids are present.

Experience is proving that for electrical installations in contaminated atmospheres all possible effort should be made to group as much apparatus as practical together in enclosed, air-conditioned rooms.

This type of protection is applicable to control, switch gear, load center transformers, motor-generator sets, rectifiers, and other similar apparatus, and equipment thus pro-

tected requires only normal maintenance. It also allows the use of standard equipment, more readily available and less costly than that of gas and watertight construction.

High-voltage power apparatus is usually of outdoor construction and not easily housed. Such equipment should be located away from corrosive atmospheres. If this is not possible, a measure of protection is provided by heavily coating all mechanical and current-carrying parts with special paint finish.

The use of totally enclosed motors, gas and watertight control and the best wiring materials available offers the only protection against corrosion where equipment must be located directly in contaminated atmospheres, providing selection of the materials used in their construction receives careful consideration relative to the corrosives to which they will be exposed.

In summary, providing clean operating conditions for equipment vital to production can hardly be overstressed if we are to guard against high maintenance cost, safety hazards, and lost production.

3. Pulp and Paper Operations Under the CMP Program, by James E. Maxwell, Business Economist, U. S. Department of Commerce.

The present CMP program is a later edition of the program in effect at the end of World War II.

Under the present program, the Defense Production Administration is charged with dividing the available supply of controlled materials. Their first responsibility is to see that the military agencies get the essential materials necessary to carry out their legitimate defense programs. Their second responsibility is to divide remaining supplies among defense supporting activities and civilian users. This division is made through claimant agencies. NPA is claimant agency before DPA for civilian activities. The division of the civilian share is carried out by NPA under terms of seven CMP regulations and 88 material control orders.

The outlook for sufficient critical materials to meet full needs of the pulp and paper industry for the next few months is not good. An indication of this is the fact that the first quarter allotment of steel, copper, and aluminum for pulp, paper, and paperboard machinery will reduce production of such machinery to approximately one half of levels prevailing in the fourth quarter of 1951. According to latest information, pulp and paper machinery manufacturers in the first quarter of 1952 will get the following percentage of their fourth quarter allotments of critical metals: stainless steel, 54%; carbon steel, 75%; alloy steel, 96%; brass mill products, 85%; wire mill products, 72%; foundry products, 82% and aluminum 82%. In view of these facts, it becomes clear that make-do and repair will again be the order of the day.

4. Substitution of Noncritical for Critical Material in the Pulp and Paper Industry, by Donald M. Platt, Project Engineer, Crown Zellerbach Corp., Camas, Wash.

The substitution of noncritical for critical materials is a timely subject and one which will, no doubt, receive increased attention in the coming months.

Before discussing substitutions, it might be well to take a look at the materials situation. The pinch, in many materials, is just beginning to hurt. For the next two years, at least, we may expect the picture to get worse in many respects. A few of the basic materials of particular interest to us which are or will be critical are: copper, aluminum, steel, and stainless steel. The copper industry is perhaps in the poorest position of any. Copper production, especially mining, cannot be increased rapidly. A drastic shortage exists now and will continue for several years. The good electrical and heat conductivity, corrosion resistance, machinability, high strength, and wear resistance of copper base alloys are properties not found to the same degree in other metals. Unlike copper the aluminum industry has expanded by leaps and bounds and, although aluminum is still a critical material, the situation is improving. Nearly all stainless steels are critical due to the acute shortages of some of the alloying elements principally nickel, molybdenum, and columbium. We may expect steel to continue short for a considerable time in spite of the recent and forthcoming tremendous increases in steel output capacity.

There are two classifications of substitution and 2 types in each class. However, only one combination is of particular interest to us—direct substitution by the user.

Perhaps the most satisfactory method of discussing the project of direct substitution of materials as it applies to our operations is by sighting specific examples:

1. Buildings—If the shortage of structural steel becomes more acute, some combinations of reinforced concrete and wood will provide very satisfactory buildings with a considerable reduction in tonnage of steel.

2. Skylights and window sash—Skylights can now be made of a corrugated plastic reinforced with fiberglass such as Alsynite or Corrolux. This material has excellent structural properties and is easy to install. This, as well as glass block, can be used as a substitute for steel or aluminum sash.

3. Bearings—There are many bearing applications throughout our mills where emergency substitution of plain bearings or special liners can be used to replace ball or roller bearings. Also with copper and bronze in short supply, we may need to look for substitutes for our plain bronze bushings and liners. It is possible to use powdered metal products for these applications. Nonmetal materials are now available and are suitable for use in many bearing applications. Examples of these are Micarta, Eelslip, Teflon, and the newer nylon and plastic type materials. Some of these may prove to be highly successful as substitutes, and many even displace metal bearings in many applications.

4. Piping—Asbestos-cement and wood pipe are the principal substitutes for steel. Rubber-lined steel is a satisfactory substitute for stainless steel in many applications. Plastics, such as Uscolite and Saran, are often suitable for small lines handling corrosives.

5. Electrical wire and cable—We may have been reluctant to change from copper to aluminum, but the time is fast approaching when we will be glad to be able to get aluminum wire and cable to meet our everyday needs. Significant improvements in the manufacture of aluminum wire for electrical use plus the design of bimetal connectors for making up aluminum to copper have eliminated most of the objections to using aluminum.

In summarizing the materials substitution problem, there are several things we should do:

1. Accept the idea of using substitutes.
2. Keep ourselves advised on the shortage situation and trends.
3. Plan ahead—design for the use of substitute materials.
4. Watch our installation jobs and our maintenance work to be assured that critical materials are not wasted.

R. M. True, *Secretary*
Pacific Section

Kalamazoo Valley

The Kalamazoo Valley Section met at the Columbia Hotel, Kalamazoo, Mich., at 6:30 p.m., Thursday, Dec. 6, 1951. Approximately 80 members and guests attended. A. T. Luey, Sutherland Paper Co., presided and introduced the speaker.

Mr. Joseph Steinbruner, Plant Manager of Chicago Branch of International Printing Inks, spoke on the subject Testing Procedures for Inks and Printing Surfaces.

A lively question and answer period followed the presenta-

tion of the talk. The questions concerned effect of moisture content of paper or paperboard on ink drying rate, effect of pH of paper on inks, possible ink adjustments on offset printing.

Mr. Steinbruner remarked that end use of product is a limiting factor in considering adjustments that may be made on inks to meet variations in printing surface. Tack of ink and composition of press rollers are also to be considered.

Mr. Steinbruner's remarks follow:

"Basic research and product development in the ink industry have resulted in a great number of improvements over the years. Inks in bygone years were basically either oleoresinous or penetrating in nature and drying was achieved by oxidation, polymerization, penetration, and evaporation. From this simple beginning, printing ink manufacture has evolved into a precision industry that is able to produce a product tailor-made for almost any printing process or end point usage of the printed piece. Resin-solvent vehicles are used that will dry under proper conditions in split seconds.

"But these improvements would not be possible were it not for the tools, the testing equipment that plays the major role in evaluating theory on a practical basis. Our factories are equipped with printing presses of various types which are used by our laboratories to check the printed result of our experimentation under conditions analogous to those existing in the pressrooms of our customers. We have single and two-color flat bed presses, single and multicolor offset presses, both web and sheet fed, a rotary typographic press which will print four colors on a web up to 1500 feet per minute, aniline equipment, and even a newspaper press of commercial speed. All of these presses have special equipment of our own design or selection that enables us to detect even minor advantages or disadvantages in one ink over another.

"No new development has any value unless it can be removed from the category of laboratory curiosities, be produced economically in quantity, and be controlled in quality. The most import factor and the one I would like to dwell on is the control that must be exercised to maintain high quality. Assuming that the ink produced on formula has been mixed and milled, a sample for testing is given to quality control. It is first checked carefully for shade by comparing it with an established standard prepared by the formulating chemist by simple drawdown. Then, by diluting the sample with from 20 to 50 times its own weight of opaque white, the chemist checks it for tinctorial strength and makes certain that the desired color value is present.

"While this is being done, manual examination of comparative viscosity has been made and a portion of the sample sent to the temperature and humidity controlled room housing our rheological instruments. We use two basic machines, the Interchemical Viscometer developed by the late Henry Green in our Research Laboratories and the Reed Inkometer developed by R. F. Reed. The I. C. Viscometer gives us the physical characteristics of the ink in dyne centimeters squared (dyne cm.^{2}) representing the tack, yield value, and coefficient of thixotropic breakdown. These values taken at relatively low rates of shear must comply with the recorded values of the standard. The Inkometer, an empirical machine, operates at high rates of shear and correlates high-speed printing conditions. Between the two we have a complete picture of whether the ink will work like the standard.

"In the meantime, bulking or specific gravity readings are taken and the dispersion examined on the fineness of grind gage. This latter device is a block of steel into which two wedge-shaped grooves have been machined. The depth ranges from 1 mil to zero and after scraping the ink over these grooves, a source of light at the proper angle will allow the quality of the dispersion to be determined by visual examination. The groove is calibrated to allow accurate recording of the fineness of grind.

"Up to this point, we have merely made a production batch of ink, controlled to our specifications. Now we must assure ourselves that the ink will meet the customer's specifications. Comparative proofs are pulled on the exact stock on which the ink is to be printed. If the exact stock is unavailable, the proofer uses the closest approximation of the printer's paper. These proofs are examined at frequent intervals to determine that the drying rate of the new lot of ink is equal to the standard. If the ink we have made will be used on printing machinery equipped with drying devices, the drying time of the ink is checked on laboratory counterparts of the production equipment.

"After the proofs are dry, they are given to a laboratory physically removed from the production and control laboratories where chemists perform any other tests required. If the printed piece is to be used for a food wrapper, soap carton, lithographed metal can, cotton bag, or any other specific printed product, the ink must be approved by this testing laboratory. Apparatus in this laboratory has been carefully selected or designed as necessary to reproduce conditions that have been experienced in field results.

"When the ink is found to have the characteristics required, it is approved for packaging. Again a sample is inspected for fineness of dispersion and this sample carefully labeled with the formula number, batch number, and date of manufacture and filed for future reference.

"More than 20 years ago, it was recognized that a thorough and continuing study of the reciprocal relationships between paper and printing ink ('paper-ink reaction' for short) would be necessary to provide a basis for further research for the improvement of printing. IPI was among the earliest in this field of investigation. A paper titled, Testing Printability of Paper with Ink, presented by F. A. Weymouth of IPI at the Annual Meeting of the Technical Association of the Pulp and Paper Industry at the Hotel Commodore in New York City in February of 1942, was probably the first successful attempt to correlate and standardize on methods of determining the relative printability of coated papers. The methods he described, by the way, have been adopted by a majority of the manufacturers of those papers. A second paper on the same subject was presented by Otto Berberich, L. F. Englehart, and Milton Zucker of the Research Laboratories to the same group at the same place six years later, based upon further studies by our laboratories in Chicago and New York. These papers were printed in the *Paper Trade Journal* of April 9, 1942, and Aug. 12, 1948, respectively.

"The testing methods described by Weymouth have been found applicable to boxboard and other packaging material and have recently been adopted by some of the board mills as well as by producers of set-up and folding cartons. Basically, there are two separate and distinct tests, one involving a visual observation comparing the ink receptivity of an unknown piece of paper against a paper of known printability by proofs pulled under controlled conditions and the second making comparisons of the known and unknown papers by proofing inks of progressively increased tack to determine the pick resistance. Please notice that in both instances, comparison against a known standard is required.

"Briefly, the printability test is performed on samples of the known and unknown which have been prepared by equalizing the caliper, and then affixing the two samples on backing piece to maintain relative position. The two pieces of board are then printed simultaneously on a precision proofing press, which has been inked with a minimum amount of ink. Because all of the contributing factors, such as ink film thickness, impression, speed of ink take-off, etc., are exactly the same, the difference in ink take-off is a true comparison of printability. An ink of low spectral reflectance will disclose such differences as wire marks, uneven surface, hard and soft spots, scabs, and pits.

"The second test, using inks of progressively increased viscosity, will reveal weakness of surface coating under condi-

ns similar to those experienced in printing conditions. This st correlates the Dennison wax method of evaluating print- surface strengths but has an obvious advantage in that does not affect thermoplastic binders frequently used in ating whereas hot wax may give erratic and misleading sults on such coatings. The tack-graded inks developed International Prinking Ink, when judiciously used, will able the board or paper manufacturer to predetermine the itability of a particular stock for single-, two-color or four- d five-color printing, by comparing the unknown against sample of known performance. This method must be rictly comparative rather than absolute since the film thick- ss of the ink, the temperature of the ink as varies by the ne allowed for mechanical distribution, the humidity, the pe of printing plate used, and the speed of pulling the proof l influence the picking.

"While it must be recognized that the testing methods are npirical, experience has proved that paper mills using the boratory proof press method of testing their product have own marked improvement in quality. Their papers are nown for superior printability.

"Because of increased interest in the production of printed aterial in gloss ink, another method of testing suitability paper for proposed end point usage has been developed. his test consists of proofing three standard inks of widely vergent composition. We use an orange ink of high pig- ent, low oil composition, a blue ink containing a small nount of pigment and a high percentage of oil, and a red high viscosity and medium oil content. Proofs of these ks can be observed visually and compared to a stock which is nown to have the property of producing jobs of high gloss.

"It is not my intention to convey to you the impression at all printing faults can be corrected by the production papers which will pass the printing tests described. We the printing ink industry do not intend to shirk our re- ponsibility in connection with the immediate production a printing job.

"Too frequently the choice of paper is dictated without the ecessary forethought that might have resulted in a superbly rinted piece. When an unsatisfactory paper is placed n roduction, the skill of a pressman can help to only a limited xtent. The inkmaker, having the greatest degree of flexi- ility, can usually adjust his product to further improve re- lts, but the best ink on poor paper will not result in a printed iece of excellent appearance any more than poor ink will sult in an excellent job on the finest paper. Good printing equires that the papermaker, inkmaker, and craftsman com- ine their skills in order to produce satisfactory printed copy ithout undue expense.

R. H. Hurst, *Secretary*
Kalamazoo Valley Section

Delaware Valley

The November meeting of the Delaware Valley Section was eld at the Engineers Club of Philadelphia on Nov. 29, 1951. pproximately 56 members and guests attended the dinner eeting. Chairman Erspamer presided.

Chairman Erspamer opened the meeting by welcoming the roup and announced that at the January meeting A. L. M. ixler of the Riegel Paper Corp. will give a talk on The De- elopment of New Products for the Paper Industry.

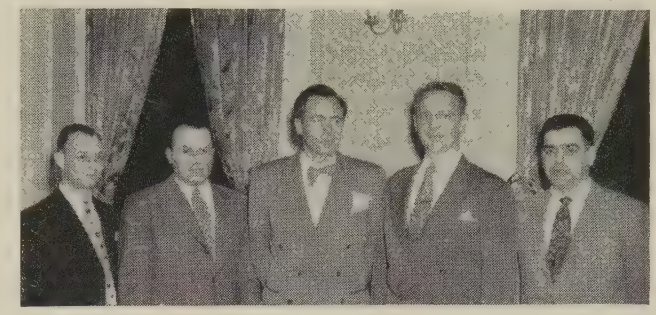
Frank Lovegren was then called upon to announce that he Albert Award letters had been circulated and that the embers were asked to give their wholehearted cooperation making this award a success.

Dr. Brill introduced the speaker of the evening, L. E. Whitmayer of the E. I. du Pont de Nemours & Co. F & F ivision. Mr. Whitmayer's subject was Color Engineering aint for Safety and Efficiency of Paper Production. His resentation follows:

Color Engineering: Paint for Safety and Efficiency of Paper Production

L. E. Whitmayer

There is a marked similarity in the growth of the industries represented here. Within the last 30 years, paint and paper making have progressed from arts to the point where they are being considered as sciences.



Officers of the Delaware Valley Section: A. J. Haug, Scott Paper Co.; A. S. Erspamer, P. H. Glatfelter Co.; H. C. Brill, E. I. du Pont de Nemours & Co.; F. J. Lovegreen, W. C. Hamilton & Sons; and E. R. Padavic, Container Corp. of America

The use of protective coatings, or paints, although sometimes confused because of the many different types and varieties, is basically for two purposes—for beauty and for usefulness. Paint jobs are expected to look well and perform a functional purpose of protecting a substrate. While the major discussion tonight will be on the function of color, let us not overlook the even more important phase of protection. For example, the wet ends of paper machines are not difficult to paint, if they are properly and adequately protected. Much more can be accomplished by applying the proper paint on the wet end of the machine when that machine is clean and dry, than can be done at a later date. Two coats of paint, in our experience, are never sufficient for protection under normal conditions; and therefore, the wet end of a machine requires more coats and more film thickness to effectively withstand the use to which the surface will be put.

In maintenance painting, it is always wise to stencil on the job the date the paint was applied and the number of coats used, so that management on its frequent inspection tours is fully conscious of repetitive maintenance costs. Such painting needs to take into consideration adequate surface preparation, application under proper drying conditions, application of a sufficient number of coats to provide adequate film thickness, and proper usage of that surface after it has been protected. You cannot bang a hammer against it and expect a paint job to last. However, paint does more than merely protect; it can also influence production in many ways through the use of color. I should perhaps say the functional use of color.

What is color? How many colors are there? What is functional color? Perhaps we better see what color is before going deeply into how color works for us.

The simplest form of color and the first one we remember is the rainbow, scientifically known as the spectrum. A ray of white light passing through a drop of rain or prism breaks up into its components or primary colors of red, green, and blue.

We must resolve color into a simplified form. All laws of color are based on primaries. Learn to describe all colors in

L. E. Whitmayer, F & F Division, E. I. du Pont de Nemours & Co., Inc., Wilmington, Del.

basic terms—hue or color, chroma or saturation, value or brightness. The physicist using lights has these three primaries—red, green, and blue. Principles of light and lighting are based on these. The colorist uses pigments, but no blend of colors will develop dark gray or black. All tinting of colors is based on these primaries. The psychologist using sensations has six primaries. His primary colors are the red, blue, green, yellow hues, black, and white. The principles of color harmony are based on these primaries. Harmony is secured with: adjacent hues, complementary hues, split complements, triads, and tetrads.

The eye recognizes a pure hue—white and black. In all the world of color there are but three basic forms of sensation. You see pure hues (red, yellow, green, blue) as one unique form and experience. You see white, which does not resemble any pure color. You see black, which again is unique, bearing no resemblance to pure hues or to white. These are the three fundamental elements.

Now, when you see a tint (pink, lavender), you say it looks like it has hue in it and white in it. When you see a shade (maroon, brown), you say it looks like it has hue in it and black in it. When you see gray, you say it is to be mixed with white and black. When you see tone (any grayish color like tan, taupe, or beige), you say it appears to have a little bit of hue, a little bit of white, and a little bit of black.

Faber Birren suggests a very simple supplement to the principles of color harmony given above by using a triangle. The following illustrates the additional basic principles.

1.	Color	White	Black	...	Harmonize
2.	White	Gray	Black	...	Harmonize
3.	Color	Tint	White	...	Harmonize
4.	Color	Shade	Black	...	Harmonize
5.	Tint	Tone	Black	...	Harmonize
6.	Shade	Tone	White	...	Harmonize
7.	Pure color	Tone	Gray	...	Harmonize
8.	Color	White	Black	Tone	Harmonize
9.	Tint	Shade	Gray	Tone	Harmonize
10.	All seven forms harmonize				

Tone is the only form of color that harmonizes with all others. A straight line through all forms of color shown on the triangle gives us an easily remembered, practical method of securing harmony. Our "Color Conditioning" colors are tones, tints, and shades; and therefore they harmonize. That is one reason why it is difficult to go off base with our "Color Conditioning" colors. All you need to remember when you strive for color harmony are two basic forms—the hue circle and the color triangle.

To get back to functional color. All this means is that color is put to work, and the work it does falls into one of three classifications:

1. Physical—That is, it is a form of energy. A white ship is cooler than a black one. Black glass absorbs heat rays. Transparent glass transmits them. Certain dyes, such as those used in our paint cleaner, change color.
2. Visual—The use of black on yellow highway signs improves legibility. The use of proper contrast eases seeing, such as our "Three-Dimensional Seeing" principles for painting machinery.
3. Emotional—Many things enter into this phase of color. An Irishman dislikes orange. This field of functional color is very complicated. Children prefer warm colors. Adults prefer cool colors.

In our "Color Conditioning" program all three phases of functional color are tied in, along with the basic principles of color harmony.

I mentioned that we are dealing with color from the psychological standpoint. One of the first aspects to consider is brightness. The du Pont Company has always made outstanding mill whites, still makes them, and still offers them for use where they are desirable; but they are not desirable in a critical seeing area. Glare and excessive contrasts tire the human eye. This being true, no colored surface within view in a given area should be more than 5:10 times brighter than any other visible surface. With this in mind, "Color Conditioning" colors were designed to fall within the 1:5 ratio. Consequently, they can be used in any combination with each other with the assurance that the eye will be unaffected by adverse seeing conditions. Functionally, it is not good to have a dado in an office because it places different ratios of brightness directly in the line of vision.

People like color. Men prefer blues, reds, and greens, in that order. Women prefer reds, blues, and greens. The dislike for violet, orange, and yellow applies to both sexes. Color preferences should be taken into consideration when painting is done. An individual can exercise his personal preference when painting his own private office, but when doing someone else's, he needs to recognize their likes and dislikes.

Another aspect is the emotional reaction to color. To most people warm colors are stimulating; cool colors are subduing. The most exciting colors are in the red and orange groups. Subduing colors are in the violet range; while tranquility is induced by the greens. Tints such as peach, buff, beige, cream, and ivory are moderately stimulating. Pale blues and violets are suppressing and should be used with caution over large areas.

Color also has warmth and coolness. I could relate many examples of this particular phase of color. Where offices are air-conditioned, sometimes a change in color alone, without doing anything to the air-conditioning system, will make the rooms appear more comfortable. Color can be utilized to make any room seem warmer or cooler simply by the considered application of paint. In practice, warm colors such as peach, beige, sunlight, etc., will make cool rooms seem warmer. In reverse, an uncomfortably warm room will seem to be made cooler by painting the walls in light blues and greens.

Appetite appeal is particularly important in color whether we consider a company cafeteria or our own dining rooms. Poorly chosen wall colors in eating places actually decrease the appetite; proper ones tend to increase it. In railroad dining cars, it was found that a lavender interior actually cut down eating time and the size of the meals ordered. In airplanes, air sickness was common where the interiors were painted in unappetizing hues. Both these situations were alleviated when proper colors were selected.

Complementary colors are used to advantage. Every color has an after-image of its complement. If, for example, you stare at a red disk for a few seconds, then gaze at any white area, you will see a blue-green disk as the after-image. Since a similar phenomenon is true for all colors, the proper knowledge of complementary colors will enable you to emphasize any given colored area by giving it a background of its complement. The human skin, for instance, is flattered by blue-green walls. Merchandise of any given color can be enhanced by supporting it with its opposite.

Why are highway signs black and yellow? Merely because of color visibility. Red is one of the first colors to lose its identity under conditions of low illumination. We use the principle of color visibility in our Safety Color Code. Alternate stripes of High-Visibility Yellow and Black are used to identify accident hazards and on the stanchions or handrails and along paper machines to indicate changes in elevation. Alert Orange is used on the interior of switch boxes and on the guard of a guillotine cutter.

Colors have size, weight, and dimensions. Why do we use black on a yellow background for highway signs? The answer is that black letters appear bigger than the corresponding yellow letters would appear on a black background. An understanding of the principles of size, dimension, and weight in color makes it possible to have small areas look larger or large areas seem smaller. Color can be applied so as to appear to raise or lower ceilings. Even tote boxes for carrying an accumulation of heavy objects can be made

parently easier to lift if they are painted yellow or light een.

Bright colors attract the eye; dark ones get less attention. is principle can be widely used in decoration or display. e bright walls of a room detract from objects within it. bdued walls, on the other hand, make objects (furniture, inters, merchandise) stand out.

The "Color Conditioning" colors are harmonious with each ner, as was pointed out from the color triangle. Since prac-ally all artificial light is colored, they are designed to give asonably good performance under all conditions of umination.

Naturally, in the time allotted to the discussion of such an -important subject, one can only adhere closely to the ndamentals. If any of you desire further information, ease do not hesitate to get in touch with us. We shall wel-me the opportunity to confer with you on your own par-ular problems. If any of you desire to use a film which we ve available, we shall be very happy to show you the Case for Color" and put it on for your supervisory group. olor like safety and prohibition cannot be regulated. Con-nuous education is required to make any of them effective.

After this splendid presentation there was a short discussion riod.

Mr. LeBeck (J. T. Ryerson & Son, Inc.): Is there a color or color scheme which you would recommend to compensate for ference in height in order to prevent accidents?

Mr. Whitmayer: Yes, in accordance with du Pont's safety lor code which is based on research and traditional association, suggest that all strike-against, stumbling, falling, and tripping zards be marked with alternate stripes of yellow and black. or example, protruding parts, curbings, low beams, railing, ading buckets and cranes, stairway approaches, etc., should be arked with this color scheme.

Mr. Schwenkler (Container Corp. of America): What is the sis of the du Pont Safety Code?

Mr. Whitmayer: These colors, yellow (high visibility), orange (alert), green (safety), red (fire protection), blue (precaution) and white and gray (traffic and housekeeping), have been selected by du Pont on the basis of research and traditional association. Some of these colors, red, green, yellow, and white, have been adopted by the American Standards Association.

Mr. Dahlgren (Pioneer Chemicals Co.): What color would you recommend for an office?

Mr. Whitmayer: An office should be every bit as pleasant and comfortable as your home. The colors used in an office, however, should depend on various conditions, e.g., if the seeing task is green, the walls and furnishings should not be green for then you have a camouflaging effect, making the seeing task harder to distinguish. On the other hand, if the contrast between the seeing task and the surroundings is extreme, e.g., if the seeing task is light and you look up at a dark object and then back at the light seeing task, momentary blindness and eyestrain result.

Mr. Tourangeau (W. C. Hamilton & Sons): From a safety angle, what color or colors would you recommend for the drier section of a paper machine?

Mr. Whitmayer: I would recommend that the drier ends be painted green or gray and the drier frames gray.

H. Wright (S. Austin Bicking Co.): In order to reduce eye-strain, what color scheme would you recommend on the end of a paper machine making a lot of colored board or paper?

Mr. Whitmayer: A neutral gray.

Mr. Rotondo (Container Corp. of America): What color should jitneys be painted from the safety standpoint and at the same time be restful for the driver?

Mr. Whitmayer: Yellow or orange with alternate stripes of black. Those parts of the jitney in the driver's line of vision may be painted some other color more restful to the driver.

Mr. Lewis (Container Corp. of America): What is your opinion of fluorescent or daylight paints in regard to safety?

Mr. Whitmayer: Most of these are not paints but only fluorescent beads. They are all right for signs where illumination is varied but are no good where illumination is constant.

Bill Marble (Riegel Paper Corp.): What paint do you recommend for damp areas?

Mr. Whitmayer: Phenolic base primer and finish paints. To do a good job, several primer coats are needed.

DONALD H. NEWCOMB, *Secretary*
Delaware Valley Section

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BROWN STOCK: Defibering and Breaking up Knots. Up to 500 tons per day. 200–300 HP.

DEFIBERING and PREREFINING: Ahead of Washers. No cutting or reduction of Freeness. Operated under pressure. Does not handicap or retard washing. Four No. 3 Claflins required for up to 600 tons per day. Knotters not required. Stock Flow: BLOW TANK—MAGNETIC SEPARATOR—PUMP—MAGNETIC SEPARATOR—CLAFLIN—WASHERS.

DEFIBERING: Ahead of bleaching. Kraft, Sulphite and Soda pulps. Deinked Stock.

PREBEATING: Washed pulps before going to the Paper Mill.

KNOTTER and SCREEN REJECTS: Kraft and Sulphite. Closed or continuous systems.

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Condensation and Hydration Reactions with Low-Grade Cellulosic Materials

EDUARD FARBER

Fields and forests continuously yield materials in which cellulose, hemicelluloses, and lignin are the main constituents. Our harvests consist of valuable grains and timber, together with large proportions of cellulosic materials of lower grade. For each 1000 bushels (30 tons) of wheat, we can reap a total amount of straw, "if cut at ground level," of 70 tons. Total production of small grain straw, in 1945, was estimated at 134.5 million tons, of which 57%, about 76 million tons, was estimated as the recoverable part of long straw (*1*). If our lumber production, as expected, reaches 36 billion board feet this year, as it did in 1942, an estimated 60 million tons of low-grade wood in form of slabs, edgings, shavings, sawdust, etc., are to be expected. Other forms of low-grade cellulosic materials are corncobs and stalks, and bagasse from sugar cane.

The availability of these materials as potential sources for more valuable products has found increasing attention in the last few decades. The present paper has the object of pointing to some recently indicated ways which have led, or may soon lead, to chemical utilizations, and which are consistent with the necessary economic considerations. The reforming into fiber boards is not included here.

A large proportion of these materials is burnt, as a way of disposal, or as a fuel. The problem of improving the use of low-grade cellulosic materials for fuel purposes has been approached from three directions:

1. The construction of the ovens has been adapted to the bulk and caloric values of these materials (*2*).
2. The cellulosic materials are compressed for greater ease of handling (*3*).
3. The fuel value has been increased by chemical pretreatments. We are here mainly concerned with this approach.

AUTO-CONDENSATION

Omitting older attempts of achieving this purpose, research in condensation reactions at high temperatures and pressures has been directed toward increasing the carbon content and, thereby, the heat value of cellulosic wastes. In the experiments of F. Bergius (*4*) and of E. Berl (*5*), water served as the carrier for the heat applied, and it acted as a solvent for the by-products. A kind of artificial coal was thus obtained. These experiments were originally undertaken to provide models of the natural process by which coal is formed and to demonstrate that cellulose was the main starting material. Berl tried to develop a commercial operation on this basis. Neither the scientific question concerning the origin of natural coal nor the economical difficulties involved in carrying out this condensation at high temperatures and pressures have so far been solved.

A partial pyrolysis of wood at temperatures below the exothermic decomposition has been investigated as to the nature of the gases and the quantities of residues formed (*6*). Since formic, acetic, and propionic acids are formed, this decomposition of wood takes place in an acidic environment. The carbon content of the solid material increased from the initial 50% (red oak) to 56.2% in 10 hours at 240°C., and to 69.5% when the wood was held at 260°C. for 10 hours.

The presence of organic acids exerts some influence, not known in its details, upon this condensation reaction.

A model for such influence is offered in experiments on condensations of glucose which are catalyzed by very small proportions of acids In this model condensation, the product is a mixture of polyoses which can be hydrolyzed back to glucose.

Glucose (dextrose) can easily be converted into condensation products with high yields. With less than 0.1% mineral acids, and at temperatures of 120 to 180°C., di- and tetramers are obtained (*7*). For example, 100 parts, by weight, of dextrose-hydrate are partially polymerized when mixed with 0.02 part of sulphuric acid and heated to 140°C. for 3 minutes. The inorganic acids are specific in their catalytic action.

Heating in the presence of small amounts of boric acid leads to higher polymers, particularly when other mineral acids or salts are added (*8*). The product obtained by heating 100 parts of anhydrous dextrose with 5 parts of finely ground metaboric acid, 5 hours at 135°C., then 1 hour at 175°C., can be fractionated into 64% of 9.4 degrees of polymerization, 14% of 6.9, and 8% of 6.5.

Acids are catalysts for hydrolytic splitting of polymeric carbohydrates when water is present in excess. Acids are condensing agents when water is excluded. Between these extremes there are those conditions of relative proportions between carbohydrate, acids, and water, under which hydrolysis and condensation can both occur. This is the case in the solutions obtained from cellulosic materials by means of concentrated hydrochloric acids (*9*).

Even with this modification, the general statement that acids cause hydrolysis in the presence of water, condensation in its absence, is too broad. The reactions are functions not only of the acid-water relationship, but, in addition, of the nature of the carbohydrates. For example, pentosans are hydrolyzed and dehydrated to furfural by dilute acids with yields varying for the different kinds of pentoses.

Wood undergoes a condensation reaction when exposed to temperatures of over 100°C. in the presence of small proportions of acids. In view of the foregoing discussion, it is not surprising that the intermolecular dehydration is only one of the reactions involved. Carbon dioxide, formic acid, aldehydes, and ketones are split off simultaneously. In this respect, the results of heating hardwoods with high pentosan content are somewhat different from those obtained with softwoods. In the main, however, the analytical proportions of cellulose to lignin are changed in the same way for both kinds of wood. The analytical lignin value increases at the expense of the carbohydrates which are originally present.

Exothermic decomposition of wood itself generally starts at temperatures of about 275°C. The acid-catalyzed condensations begin much below this temperature. When 2 grams of sulphuric acid are added to 100 grams of comminuted wood (medium coarse sawdust), temperatures of about 200°C. and reaction times of about 30 minutes are sufficient to yield a converted sawdust, in which hydrolyzable carbohydrates are greatly reduced, while nonhydrolyzable "lignin" is considerably increased. Sawdust from Douglas-fir, heated with 2% sulphuric acid in 1 hour to 190°C. and kept at this temperature for 30 minutes, gave, upon analysis by treatment with sulphuric acid of 72%, a "lignin" value of 81%, and 22% "glucose." Sawdust from a mixture of hardwoods, mainly maple and yellow poplar, under otherwise the same conditions of reactions, was converted into a product giving 50% "lignin" and 34.6% "glucose."

By increasing the proportion of acid lower temperatures can be used for similar results (*9a*).

Such condensation products from sawdust, which could also be produced from other low-grade cellulose materials, have shown beneficial effects as additions to soil. In preliminary experiments, added in amounts corresponding to 10 tons per acre to a sandy soil, they promoted the growth of radishes, beets, tomatoes, onions, etc.

The idea of using lignin for soil improvement is not new. The residue from wood hydrolysis has been suggested for this purpose. The waste liquor from the sulphite pulping process seemed to offer an inviting application in this field. It has definite effects on soil particle aggregation (*10*). For field

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lants, some adverse results have been reported, but beneficial ffects have also been published (*11*).

The effects of mulches (*12*) and organic soil amendments are o remarkable that they deserve a great deal more of scientific esearch.

Numerous patents have been issued concerning the use of ellulosic wastes as carriers for nitrogen. Reactions at very igh temperatures (over 300°C.) (*13*) are proposed for this urpose.

HETERO-CONDENSATIONS

As temperatures in treating cellulosic materials reach 300° C., condensation approaches the range of charcoal formation. The presence of other substances, capable of reacting with the ellulosic substances before they break down into "charcoal," an change this course of reaction completely. Alcohols or henols react predominantly with the lignin when they are sed together with water (*14*). Methods of producing cellulose have been based on this elective reaction. In the absence of water, and with acids as catalysts, the entire wood ubstance reacts and is changed into resinous materials. The nfluence of water is as pronounced and complex as in the ydrolysis and condensation equilibria mentioned before. Wood and other cellulosic materials dissolve under reaction n phenol or cresol, when heated with the addition of a little nineral acid (*15*). With loss of water and some volatile byroducts, resins are obtained which are thermoplastic up to at east 200°C.

Acetylation of wood can be achieved under conditions simiar to those for cellulose. Reaction with the resinous and atty acids contained in tall oil, or with the abietic acids of vood rosin, begins at temperatures of about 300°C. for resilues from furfural production, and may require up to 360°C. or wood (*16*).

Isolated lignin, obtained after hydrolysis of wood, is even nore reactive (*17*).

The resins produced by this condensation with tall oil or osin are primarily thermoplastic. They react with boric cid at temperatures of about 200°C. to form gels. Boric cid is a versatile condensing agent; its effect on pinewood osin consists in increasing its melting point after prolonged eating to 100 to 250°C. (*18*). Small amounts of hexamethylnetetramine at high temperatures produce gradual further ondensation, up to an infusible and insoluble state (*19*). In his respect, these resins from cellulosic materials resemble henol-formaldehyde resins, while they share with rosin the eactivity toward boric acid.

TOPOCHEMICAL HYDRATION

The chemical reactivity of cellulosic materials is greatly nfluenced by structure. The fiber of plant cells provides not nly mechanical strength, but also chemical stability. By lestroying this structure, the chemical stability is greatly educed. Finely ground wood reacts with much greater ase than coarse wood particles. Beechwood, reduced by lry grinding to an average partial size of 1 to 2 mu, can be nethylated in one step to a methozyl content of 36% OMe, nd acetylation with acetic anhydride in pyridine gives a roduct containing 40% COMe (*20*). The crystallinerdered state of cellulose disappears upon grinding it to fibrils f 100 to 750 A thickness (*21*).

Cellulose is deeply affected by fine-grinding. After grindng cotton cellulose in a Wiley mill, the loss of degree of rystallinity was found to be 3% when a 2-mm. sieve was used, % with an 0.84-mm. sieve, and 17% when fineness was inreased to about 0.2 mm. (*22*).

Some of the controversy concerning chemical bonds beween lignin and cellulose can be explained by considering the orm of cellulosic material which was investigated.

Besides, water again is of decisive influence. Fine grinding n the presence of much water can be carried beyond the primary fiber structure to a stage of high dispersion. Cellulosic materials of high pentosan content, like most straws and hardwoods, are more readily changed by wet grinding than low-pentosan softwoods. Papermakers have used this in producing glassine paper which resembles the uniform structure of cellulose regenerated from solutions of its compounds. The glassine effect does not depend on high cellulose content of the paper finish; pulp produced without material removal of lignin from bagasse or hardwood can be similarly converted.

It has been attempted to utilize such hydrating processes for increasing the digestibility of woody substances (*23*).

Continued grinding of cellulosic materials in water leads to a peculiar physical state which deserves the name colloidal in its original meaning. In this state, the cellulosic materials can be used as glues, particularly for paper (*24*) or for the production of wallboard (*25*).

MOLECULAR HYDRATION

Topochemical hydration does not increase the reducing action, measured for example by the copper number, the real solubility, or ion adsorption. Soluble anhydrous carbohydrates are produced by the action of concentrated hydrofluoric acid, while sulphuric or hydrochloric acid dissolve cellulose and hemicelluloses by hydrolytic action. However, when dilute hydrochloric acid of 3 to 8% is used at temperatures of 130 to 180°C., a considerable part of the carbohydrates is converted into levulinic acid. Normally only a small by-product of hydrolysis, levulinic acid is, under these conditions, obtained in yields of 16% of pinewood (*26*). The lignin is left undissolved, although not unchanged. Compounds of lignin with sulphuric or hydrochloric acid are formed.

Such compounds are particularly important when sulphurous acid is used. Solutions of the free acid hydrolyze the carbohydrate part of cellulosic materials without solubilizing the lignins (*27*). Partial neutralization of the sulphurous acid leads to solubilization of the lignin without attack on the cellulose. Complete neutralization reduces the reactivity with lignin so that neutral sulphite solutions are used for semichemical pulping. An excess of sodium hydroxide over the neutralization equivalent makes it possible to carry out a mild reaction with hardwoods to prepare them for pulping (*28*).

The specific reaction of dilute solutions of alkalies is used for removing lignin from straw (*29*), or from wood. Under more violent reaction conditions, by fusion with an excess of sodium or potassium hydroxide, at temperatures up to 250°C., oxalic acid is the main product from wood. Between the mild reaction which leads to cellulose, and the violent reaction which produces oxalic acid, there is a medium range in which cellulosic materials are converted into alkali-soluble resins. Either wood or lignin (*30*) is heated with an equal quantity of sodium hydroxide, in the practical absence of water, to temperatures of about 200°C. The addition of small amounts of sulphur prevents charring. Zinc oxide also acts as a stabilizer. The reaction proceeds rapidly once the temperature of about 200°C. is reached. The cellulosic material becomes completely soluble when water is added to the mixture. Acidification precipitates a dark-brown resin in yields of 80 to 85% when lignin was used, somewhat less when wood was the starting material. Oxalic acid is among the by-products of this reaction. The main part of cellulosic materials can thus be converted into an alcohol and acetone-soluble resinous acid.

CONCLUSION

The described methods of condensation and hydration reactions with cellulosic materials offer a variety of possibilities for chemically utilizing low-grade by-products obtained from trees and agricultural crops. In view of the great quantities in which these by-products are available, it is an advantage to have many reactions and products from which to select those best adapted to specific situations. Further chemical research in fundamental and in technological directions will be

necessary and desirable to develop the possibilities offered by these reactions.

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Military Barrier Materials

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Barrier materials and the barrier material industry came into prominence during World War II. These materials were designed to prevent deterioration of a large number of military items during transportation, handling, and storage in the various theaters of operations throughout the world. Military barrier materials may be roughly segregated into three groups, i.e., greaseproof, waterproof, and water-vaporproof. One or more combinations of these materials may be used to package a large variety of items that require protection against damage caused by corrosion.

Greaseproof barrier material is intended for wrapping metal parts and assemblies which are previously coated with a corrosion-preventive compound. This material is intended for initial wrapping of parts, prior to additional packaging. Specifications JAN-B-121 prescribes two grades of material, i.e., grades A and C. Grade A material is indicated where a noncorrosive barrier, which does not require sealing, is desired for retention of a preservative on the item. Grade C material is for use where a noncorrosive, moldable, sealable, and self-adhering barrier material is desired. This material can be molded around a part, or around a container, and then suitably sealed by dipping in hot wax.

Waterproof barrier material was designed to provide waterproof protection for various items and is used as a case liner, interior wrap, temporary tarpaulin, baling wrap, etc. Case liners are usually furnished as prefabricated or "tailor-made" bags with joints, seams, and closures sealed with adhesives or by other suitable means. This material is also used as an interior wrap to protect individual packages or assemblies against exposure to water. Interior wraps are used to cover material packed in fully sheathed crates thus protecting them against penetration of water. In these applications the shrouds act as a watershed and are so constructed that they do not interfere with the free circulation of air around the item packed. This material is also used as a crate liner and is usually inserted between the sheathing of the crate and the inner diagonals, struts, or braces.

Water-vaporproof barrier material is used to protect items which cannot be coated with a preservative requiring removal prior to the use of the item. These items comprise equipment or materiel, mechanical or electrical, including assemblies and partial assemblies having close tolerances and finished parts susceptible to deterioration in a humid atmosphere. This material is also used to pack equipment which cannot be coated with a corrosion preventive because of the complexity of the item. This material is recommended for floating bag applications, carton-barrier carton applications, and packaging of cushioned items in the barrier bags. The material is intended for use in conjunction with a desiccant which is required to remove the residual moisture from the air to prevent the accumulation of moisture within the package.

To provide background for this discussion it is believed desirable to briefly outline some of the handling and storage conditions encountered by military materiel. Storage and handling conditions, to which military materiel is subjected during wartime, are generally much more severe than those considered by the average packaging engineer engaged in design of packing for commercial items. Many packages designed for shipment within the continental United States or overseas arrive in perfect condition when shipped by commercial carrier. However, if these packages were forwarded to some distant point under battle conditions, they would probably arrive at their destination but would be a candidate for the scrap pile before distribution could be accomplished. To a soldier in wartime this is a very serious problem since his ability to win a battle depends largely on the adequacy of his equipment. Military experience shows that there never has been a group that requires the toughest of packages than the personnel used for unloading ships at overseas ports under wartime conditions. Much indigent labor is used, and the language barrier makes it difficult to train these laborers. In many instances a box is dropped from a man's shoulder or from the platform of a truck during the handling and unloading operations. This type of rough handling, together with the deterioration caused by poor storage conditions, poses a serious problem for military packaging engineers. Available data show that, at the beginning of World War II, 60 to 75% of certain packages shipped overseas were either badly damaged or damaged beyond repair. It was necessary to set up rehabilitation centers for reworking and repacking the materiel. This was extremely costly and seriously hampered the overseas operations. The military personnel who investigated this condition realized that the design of packing for domestic shipment was inadequate for overseas shipment and recommended that steps be immediately taken to redesign all packaging and packing. Approximately in 1942, steps were taken to revise the military packaging program and to establish packaging laboratories and provide more rigid tests and requirements. The redesign program progressed rapidly and losses caused by inadequate packing were reduced substantially.

In the establishment of requirements consideration was given to the fact that storage conditions for military materiel may be extremely poor since it is not always possible to ob-

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tain adequate storage facilities. Many items require transportation and storage on beachheads, in steaming jungles, in the frozen north, and in many other places which impose conditions entirely foreign to the conditions encountered in usual overseas shipments. Based on these conditions, it has been determined that a large portion of the packs currently designed must meet transportation and storage conditions of 160°F. and −65°F. Consideration must also be given to storage at −80°F. for those items which will deteriorate under this condition. These limits are considered to be extreme but it is possible for a package to be subjected to one or all of these conditions.

Grade A greaseproof barrier materials used during World War II were mainly acetate film and kraft construction, and various coatings on kraft paper. These constructions were intended to be greaseproof and noncorrosive, but in many instances, were neither.

The construction used for waterproof barrier materials usually consisted of kraft paper-asphalt laminations, and were considered to be waterproof for a minimum of 4 hours. This construction allowed exudation of asphalt at elevated temperatures and generally cracked when creased at freezing temperatures. Since the military was not able to control the rainfall to 4 hours, nor able to predict at what temperatures the materials would be subjected, it is obvious that the materials were not entirely satisfactory.

One of the combinations that was used in large quantity for water-vaporproof barrier materials consisted of polyvinyl butyral coating on lead foil, laminated with asphalt to kraft paper, which in turn was sometimes laminated to a scrim cloth. This material was heavy, had poor high and low temperature characteristics, and was considered expensive. It was effective for certain shipments provided the barrier materials were not subjected to extreme temperature conditions and rough handling was held to a minimum. Since this material exuded asphalt at high temperature and cracked at low temperature it was obvious that improved materials should be developed.

Since the end of World War II considerable effort has been expended in improving this barrier material and specifications covering the material. It is true that certain barrier materials have been greatly improved since World War II, however, examination of the records and the results of numerous tests show that much research and development should be accomplished to improve the barrier materials that are currently being purchased and used by the military services.

It has been stated that military specifications have not kept pace with industry's development of improved materials. It has also been stated that the Government has not cooperated with industry in the development of these specifications. That is not a true or accurate statement. From the date of initiation of this development program barrier material manufacturers have been asked to cooperate and assist us in the development of these materials. These manufacturers have been very generous, and a considerable amount of thanks should be given to those companies that offered us their technical skill and "know-how." Without this help the program would have been extremely difficult, and our progress would have been much slower. It is believed that most barrier material manufacturers are in agreement with the development program as currently established and the assistance given by industry in the program is sincerely appreciated.

Before discussing each of the three specifications individually, it is believed desirable to briefly discuss several of the new materials which are being used in connection with the development of improved barrier materials. Probably the most important and the most promising material being used is polyethylene. This material is being considered for all three types of barrier material. It is particularly suited for barrier materials because of its low temperature flexibility, chemical inertness, lack of odor, nontoxicity, ability to age, heat-sealing characteristics, and other qualities, well known in the packaging field. Various techniques for laminating polyethylene film and applying polyethylene coatings to paper have been developed, and one of the important methods is known as the hot melt process for coating directly on paper. Polyethylene is probably best known for its commercial uses such as lining for tobacco packages, fiber drum liners, and multiwall bags. Polyethylene coated paper is used for protecting many moisture-sensitive commodities such as fertilizers, calcium chloride, caustic soda, powdered milk, quicklime, and other similar items.

Another material which is currently being developed and is proving to be extremely satisfactory is a plasticized vinyl film. This film is proving to have excellent aging characteristics and remains flexible at −65°F. It is believed that these low temperature vinyl films will offer competition to polyethylene in many moistureproof barrier applications.

There are several other films currently being investigated that offer excellent possibilities for use in barrier materials since these films also have good low temperature flexibility. These films are undergoing development tests at Picatinny Arsenal, however, the data available to date are not conclusive.

JAN-B-121

The first specification for discussion is JAN-B-121, Greaseproof Barrier Material. This specification currently covers two grades and three types. As previously mentioned, grade A is a nonsealable wrap for preservative retention and grade C is moldable and sealable for a similar purpose. The types indicate the weight or strength of the material, i.e., heavy duty, medium heavy duty, and light duty. It has been recommended that this specification be changed to prescribe one type. This recommendation was made because, in many instances, the personnel hired for packaging operations will use a type III in lieu of a type I if the type III happens to be nearer than the required type I. It is realized that we shall be accused of overpackaging. However, it is the opinion of many packing specialists that it is better to overpackage in some instances than to run the risk of inadequate packaging in other instances with a net result of complete loss of a portion of the material packaged. For that reason, one heavy-duty type of barrier material is being considered.

Since this specification covers a greaseproof material, the tests for greaseproofness are of primary importance. The test currently prescribed in the specification is a 900-second turpentine test, and it appears that this test is insufficient since turpentine is not a grease and 900 seconds is a very short time compared to the storage life expected for many items. The turpentine test is considered as not a greaseproofness test, and is in reality, a pinhole test. This specification also states that the greaseproof material shall be noncorrosive, and outlines tests that were intended to control this feature. It has a test to determine the acid content and states that the water-soluble acidity of the finished barrier material shall be not more than 0.02% equivalent sulphur trioxide. This test is believed to be a good test and is expected to remain in a revision of the specification. Another test which is considered to have a bearing on the corrosion characteristics of a barrier is the pH or hydrogen ion concentration test which states that the finished barrier material shall have a pH of of not less than 6.5 nor more than 7.5. Since this is a pH value of the entire sheet, it is considered an unsatisfactory test. It is known that in some instances, manufacturers have used laminated barriers that had one sheet highly basic, and other sheets highly acid, with the resulting pH value of 7. It is also known that where the barrier was basic, acid has been added to neutralize the composite sheet. There is a large range of acids that might be used, some of which have a tendency to migrate to the surface during aging and seriously injure the strength of the barrier and cause corrosion of the packaged part. It has been argued that a higher pH

might not necessarily be detrimental, and certain sheets have been manufactured with a high pH since the sheets have a tendency to lower in pH during aging. This condition can also be serious as no one can forecast the extent of reduction in pH value. Since a pH value in a barrier material can be manufactured like a cook's broth and the face of the sheet in contact with the item to be protected might be highly acid, it does not appear reasonable to use a test where the composite is used in determination of the pH. It has been suggested that the pH test be established on a basis of testing each individual ply after removal from the remainder of the sheet. This type of test has been given consideration, but it is believed that it would be unsatisfactory because in many instances it would be impossible to separate the plies. A series of development tests to overcome these deficiencies have been conducted at Picatinny Arsenal, and the results of these tests are being tabulated for use as a basis for a proposed revision to Specification JAN-B-121.

It is planned that this specification will cover one type, as previously mentioned, and two grades. The grades will be the current A and C, and each grade shall have two classes. Grade A, class I, will be a heat-sealable structure having more than one ply (plastic-coated kraft paper or foil is considered as two plies). Class II will be a heat-sealable, unsupported film such as polyethylene, vinyl, vinyl polymer, vinyl copolymer, or other similar materials. The type I material will be used in sheet or fabricated bag form for various packaging operations, and the class II material will be used for greaseproof prewrapping or in fabricated bag form. The grade C will have two classes, I and II, i.e., one class with a self-adhering coating applied on the nongreaseproof side only and the other a self-adhering coating applied on both sides. The tests outlined in the following paragraphs are being conducted for development purposes and it is expected that only a portion of the tests will be included in the specification.

A group of 18 tests are being conducted in the development of the specification and these tests are as follows: A flexibility and creasing test at a low temperature of −65°F. To comply with this requirement the material must be able to withstand flexing and creasing immediately after storage at −65°F. without delamination, pinholes, or other deleterious effects. The second test consists of an aging stability test and materials are being stored for 1 month in which 15 days are at 160°F. dry and 15 days at 160°F. with a humid atmosphere (simulating desert and tropic conditions). To date, many of the materials have proved their ability to withstand this type of aging test. The third test is a greaseproofness test. Several greaseproofness tests are under consideration and the one which appears most satisfactory will be used. It is expected that the duration of the test will be a minimum of 24 hours. One test provides creasing immediately after storage at −65° F. and testing in contact with a specification preservative oil or grease at 160°F. Other development tests are continuing in which homogeneous mixtures of diluents and greases or oils are applied to the material in an effort to develop as severe a greaseproofness test as possible and utilize the best currently available materials. It is the opinion of the personnel developing the tests that a greaseproofness test based on specification greases or oils currently used by the military services will provide an effective test. The fourth test is a water resistance test which is applied to the material after low temperature flexing and creasing and the material shall be resistant to water for at least 16 hours. The question will probably be raised as to the reason for a water resistance test for greaseproof barrier material. This test is being considered since results of tests have shown that some of the currently available greaseproof coatings are completely water soluble. This test is also being added to provide that a material which meets type I, grade A of this specification may be used for interior packaging bags. The fifth test is an oil absorbency test. The amount of oil absorbency which might be tolerated has not been established, but it is believed that approximately 0.25 gram per square foot when exposed for 16 hours at 73°F. and 50% R.H. may be used in connection with this test. It is considered that penetration tests do not indicate the amount of oil absorbed by a barrier material wrap, and oil absorption may be as objectionable as oil penetration in decreasing the value of a protective oil film. The sixth test is a corrosion resistance test. This test is one in which the barrier material is placed in contact with a polished steel cylinder and then placed for a minimum of 3 days at various elevated temperatures and 95% R.H. This test is being developed since it is believed that although the surface pH and total acidity may be important factors in corrosion, they do not in themselves indicate the corrosiveness of a wrapper because of the appreciable effect of other factors. Test results show that surface pH, acidity, and water vapor transmission rate have an influence on corrosion. Composition of some barriers is such that high temperature and high humidity conditions cause a marked change in pH, therefore the original pH determination is of little value in predicting the corrosiveness for this type of barrier. Additional tests in connection with this corrosion resistance problem are being developed and results to date are inconclusive. The seventh test is a blocking test in which grade A material shall not block at 160°F. and grade C material shall not block at 120°F. It is believed that this test will provide a more satisfactory material since blocking is a cause for failure of barrier material by tearing at or around a blocked area during transportation. Little difficulty is encountered in obtaining grade A and grade C material to meet these requirements, and work is continuing to develop a grade C material having a higher blocking temperature. The eighth test is a seam strength test. This test is being considered since a bag is only as good as the seam with which the bag was sealed. The ninth test is for oil resistance of the seam. This test is being added since a good greaseproof barrier should also have oil resistant seams. The test will be conducted at 160°F. and the seam shall be resistant to oil for at least 24 hours. The tenth test is a water resistance test of the converters seams, and it is considered that the seams shall be waterproof for a minimum of 16 hours. The eleventh test is the acid content test as previously described. The twelfth test is a chloride content test. This test is being included since it is known that an excessive amount of chlorides in a barrier material have a definite corrosive effect on certain metals. The minimum content for inclusion in a revision of the specification has not been established, but it appears that 0.02% maximum may be the figure used. The thirteenth test is a pH (hydrogen ion) test. This test is being considered primarily as an identification test. Tests are being conducted to determine whether the requirements may be liberalized to 7 ± 1 or greater instead of the usual $7 \pm 1/2$. The other five tests are bursting strength tensile strength, tear resistance, spring back, and free stretch. This last test is being added since the present specification states that a stretchable material shall have a stretch of not less than 15%, but fails to state that the material after stretching 15%, shall not show evidence of delamination or other deterioration. Many of the tests listed are for grade A only and it is believed desirable at this point to outline the differences between the proposed tests for the grade A and grade C barrier materials. The flexibility test for grade C is at −10°F. in lieu of −65°F., and the following tests do not apply to grade C: seam strength test, oil resistance of the seam, water resistance of the seam, and the free stretch.

JAN-P-125

The next specification for discussion is JAN-P-125 covering waterproof flexible barrier material. This specification prescribes kraft paper-asphalt laminations in three to five plies alternating the paper and the asphalt. The specification currently prescribes 15 types which cover the various weights of paper, weights of asphalt, and number of plies. Since very little information is available regarding the exact application

for each type, considerable confusion exists when a type must be selected for a specific application. Kraft paper-asphalt laminations are generally unsuitable for case liner and similar applications. In most instances, the asphalt exudes at 160°F., cracks at −65°F., and has a water resistance of from 4 to 12 hours. Data available on field performance show that case liners and similar applications have failed in many instances because of soaking through, puncturing, or abrasion. When this condition occurred near the top of the container, water was allowed to enter and remain within the liner thereby causing damage to the items packed.

Examination of the current specification showed the reasons why failures occurred when the barrier material is used for military packaging. First, the material is not a satisfactory material for use under extreme temperature conditions. Second, the tests comprise of only a tensile strength, a stretch, a minimum water resistance test, and an ineffective flexibility test.

All of the details of the proposed revision to the specification have not been established to date and considerable more development work is indicated before this may be accomplished. The results, to date, indicate that the modified specification will provide for four main classes, and only one type for the reasons previously outlined. The first will cover a wrapping classification which shall be nonreinforced and nonstretchable; the second, a case liner application which shall provide for two subclasses. One subclass will cover nonreinforced and nonstretchable materials utilizing both heat-sealable and nonheat-sealable construction. The other will cover nonreinforced and stretchable in one and two directions and also utilize heat-sealable and nonsealable material. These two subclasses of material will be predicated on definite load limits to be established for field use. The third class will be a baling classification utilizing reinforced pliable material and nonreinforced (one-way and two-way stretch) material. The fourth classification will be for temporary tarpaulins utilizing reinforced pliable material. It is considered that these four classifications will provide adequate material for all of the waterproof barrier requirements and eliminate a number of types which have never been clearly defined for use.

The problem of improving this specification has been approached by conducting tests for the purpose of obtaining data that might be correlated with physical characteristics tests. It was decided that service conditions in the field should be reproduced as nearly as possible by testing procedures. Since waterproof barrier material is used extensively in case liners it was decided to conduct the initial tests on liners.

A case liner, when in a box that is distorted under load, is subjected to a combination of stresses that is not produced when the case liner material is subjected to the ordinary physical tests in a laboratory. It is believed that the effect of this action on case liners can be determined, and a set of physical tests devised to produce an equivalent action. It was decided to use as a basis for correlating these tests, one half of the performance test as outlined in Specification MIL-B-131A. In this test, the box containing the case liner is subjected to storage at 160 and −65°F., tap water spray, and rough handling under all of these conditions. The results of these tests will be correlated with the physical tests for determination of the optimum physical tests.

Fifteen tentative physical tests have been considered for development of the proposed specification for waterproof barrier material. The material will be subjected to an aging test at 160°F. and 60% R.H. for approximately 15 days. After this aging test, the material will be creased and flexed at −65°F. After creasing and flexing at −65°F. the material shall show satisfactory water resistance for at least 100 hours. The water vapor transmission rate has not been established to date, but it is believed that it should be a low rate. A wet and dry tensile strength and a bursting strength test will be conducted. A puncture test for this type of material is being developed. However, the exact nature of this test has not been established. An abrasion resistance test conducted under both wet and dry conditions is also being developed. A flexibility test at 0°C. is being developed for identification purposes only. The material shall not block at 160°F. and the material shall show no bleeding or staining when tested at 160°F. It is also proposed to incorporate a stretch test using one- and two-way stretch and a seam-strength test. The material shall be resistant to mildew and similar microorganism deterioration. It is believed that results of these tests will provide sufficient data for a specification which will assure that an adequate waterproof barrier material will be available for military applications under tropic, temperate, and arctic weather conditions.

MIL-B-131A

The third barrier material specification for review is MIL-B-131A covering flexible water-vaporproof barrier material. This specification currently provides a material which is adequate for all theaters of operations. However, it is considered that the ultimate has not been reached, and considerable research and development work is being accomplished to improve this specification. As a result of this work and intensive testing of many combinations submitted by laminators of water-vaporproof barrier material, the materials approved under that specification show a distinct improvement over the materials available a few years ago. The currently acceptable barrier material for Class A application usually consists of a vinyl-coated aluminum foil or vinyl film laminated to aluminum foil to which is laminated polyethylene film and backed with a cotton scrim cloth for reinforcement. This current barrier material is much stronger, more uniform in heat-sealing characteristics, much lighter in weight and more pliable and durable, particularly in regard to low temperature handling, than the materials purchased under previous revisions of this specification. The material, in spite of the fact that it is far superior to previous materials, is also lower in cost. One of the basic materials which has made this combination so successful is polyethylene. This material, having an extremely good low temperature flexibility, is particularly applicable to a low temperature flexible barrier material. It should be understood, however, that a combination of this type probably could not have been developed during World War II, because polyethylene film was not readily available and because of extreme limitations on aluminum foil.

This material is an excellent illustration of the development which can be accomplished through experimentation with a wide choice of components, and combining of the "know-how" of the barrier industry and the Government laboratories. This improved barrier material is being manufactured by many companies and is being used for the packing of a large number of types of war material, ranging from aircraft engines to many electronic devices.

The current specification bases the qualification of the material primarily on a performance test, in which the material is fabricated into bags and tested in chambers or cabinets providing temperature conditions ranging from −65 to +160°F. It is also placed under simulated rain-making equipment for certain stipulated intervals and rough handled in a hexagonal box testing drum, 14 feet in diameter, and of a type generally in use in box testing laboratories. This type of performance test simulates as nearly as possible the actual conditions encountered in the field and is probably the most practical method of testing to date. However, this test is considered to be not entirely reproducible over a small number of tests, is time consuming, and is not readily adaptable to check testing in a manufacturer's plant. It is believed that it will be possible to substitute a series of

aging, flexing, corrosion, and delamination tests for the performance test currently specified.

In an attempt to provide improved tests and equipment, development tests are being conducted on new types of low temperature flexing equipment. The experimental tests are being conducted at temperatures ranging as low as −65°F. One of the flexing machines being considered is similar to a machine which was developed at the Naval Air Experiment Station in Philadelphia. This machine utilizes a hollow cylinder of water-vaporproof barrier material, $3^1/_2$ inches in diameter and 8 inches long. The cylinder is twisted through an angle of 450° during the first 3.6 inches of axial travel of a 6-inch cycle. Preliminary test results show that considerable difficulty is encountered in the application of a 450° twist on a hollow cylinder of barrier material at extreme low temperatures by helical action with a fixed pin. It is proposed to modify the 450° twist or eliminate it completely for flexing tests conducted at −65°F. Another proposal is to substitute a flexing action whereby the barrier material will be subjected to an accordion flex. Tests are currently being conducted using the accordion flex, and the results appear to be satisfactory. The sample materials will be flexed a varying number of times at low temperatures, and after the flexing tests, the WVT rate of the flexed material will be determined. Concurrently, the tests are being conducted on unaged material and additional materials are being aged for 7-day periods ranging up to 2 months. The aging cycle comprises 16 hours at 160°F. (dry) and 8 hours at 100°F. and 95% R.H. Samples of materials are withdrawn from the aging cycle at intervals of 1 week, flexed on the flexing machine, and then an WVT rate is determined. A curve of WVT rate versus the number of days aged is being plotted, and when these data are compiled it is hoped that a definite aging test can be determined for incorporation in a revision of the specification.

A folding endurance machine is also being developed. This machine has an oscillating head provided with clamping devices to apply tension on the samples of barrier material being folded. The oscillating movement of the head is such as to fold the barrier through an angle of 135°, both right and left of the position of zero fold. The folding tests are conducted at various low temperatures. This machine is being developed since good folding characteristics of barrier materials at low temperature are among the requirements for an adequate barrier material. Results of preliminary tests indicate that materials which have been satisfactory in the performance tests do not generate pinholes up to 1000 flexes. However, those materials which were unsatisfactory in the performance tests, showed a tendency to pinhole very rapidly at approximately 200 to 300 flexes.

The results of tests conducted in accordance with the current specification and on the proposed equipment will be studied, and those tests which appear to provide the most consistent results will be selected for inclusion in the specification.

A new proposed specification has been prepared to control the manufacture of bags fabricated from materials qualified in accordance with specification MIL-B-131A. The specification covers the preparation of all bags, pouches, hoods, and sleeves which are fabricated from this material. The specification comprises four tests and requirements as follows:

1. All seals and seams shall be not less than $^1/_2$ inch wide and shall be continuous without blisters, delamination, or wrinkles.
2. All seals and seams shall not allow separation or delamination of the heat-sealed area when tested with a 10-ounce load at 160°F. and a 2-pound load at 100°F. for a period of one hour.
3. All seals and seams shall withstand a water penetration test of 24 hours.
4. All fabricated seals and seams shall pass an oil penetration test for 24 hours at 160°F.

Development tests are being conducted for detecting pinholes in the barrier material. It is planned to include a test in the specification by which the material shall be fabricated into the shape of a shallow pan with the backing on the outside of the pan. This pan shall be partially filled with a mixture of water and dye incorporating a wetting agent to provide rapid penetration through any pinholes. The material for the test shall be cut across the seams using side, bottom, and center seams, if applicable. The pan shall then be formed to provide a seam equidistant from each side. This procedure allows for test of the seam and the material and will detect excessive rough handling of the material during the fabrication of the bags.

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Preventive Practices in Electrical Inspection

H. R. MEYER

Just as a means of providing a new approach, and for today only, I would like, with your permission, to change the name of your association to Technical Association for Preventive Practices in Inspection Work. This should in no way detract from the dignity of your group and may add to the thought uppermost in all minds, and that is—how to best insure continuous operation of a machine where any down time means direct loss of revenue from the available business on hand.

Once again, in a span of 10 years, we are confronted with a situation, which, although not of our own individual choice or making, has brought us to a point where, unless we eliminate our prodigal waste of natural resources, we will end up as another dependent nation. The fracas partially suspended in 1945 should have taught us all a lesson regarding the necessity of preserving what we have. Under the governmental regime, which, for the past 18 years, we have had to labor, the watchword has been "spend and waste." Very shortly, unless we change our mode of thinking, we will have nothing to waste.

One of the best examples of what can happen to a natural resource can be found up on the Mesabi Range in northern Minnesota. One look at those great holes in the ground with an understanding of the fact that practically all of the so-called soft iron ore is gone, soon brings one to a realization of what is happening. To bring the subject closer to home you know what was happening to our lumber supply before we got smart with a replanting program. All of which brings the sobering thought that anything which will help conservation should be analyzed. Maintenance will do more toward this goal than almost any one element of our economy.

Among other things, I have been asked to discuss, to a limited extent, the electronic elements used in paper mill drives. When you come right down to it the theory of the electronic tube is not new. In fact, there is really nothing new in this universe of ours. We are day by day getting a little smarter and finding out more and more about things that have always existed. Actually, manufacturing as it is carried on today, can be likened to the simple four-pronged valve or vacuum tube that was used in our first radios back in the 1920's. (See Figs. 1 to 6.)

In the construction of the vacuum valve or the simple electronic tube we have first the factory, represented by the glass or metal envelope. We then set up a management organization which, in the tube, we will call the plate or pressure element. Obviously, management alone is not productive so we add the worker and tool element, in our case the filament or emission medium. We now have two parts of our organ-

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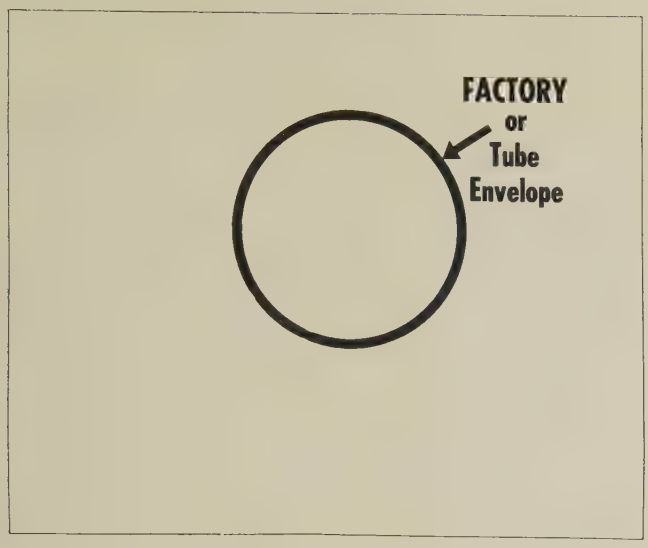
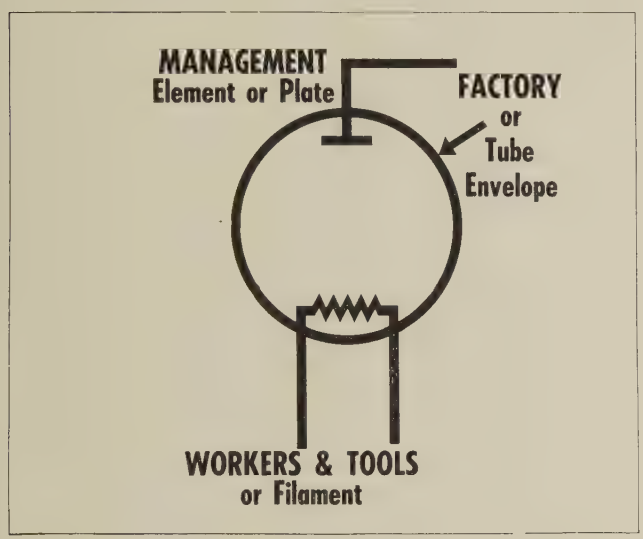
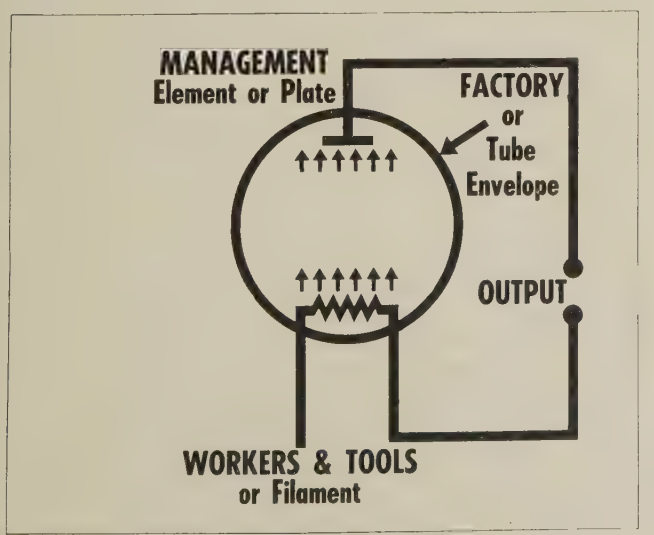


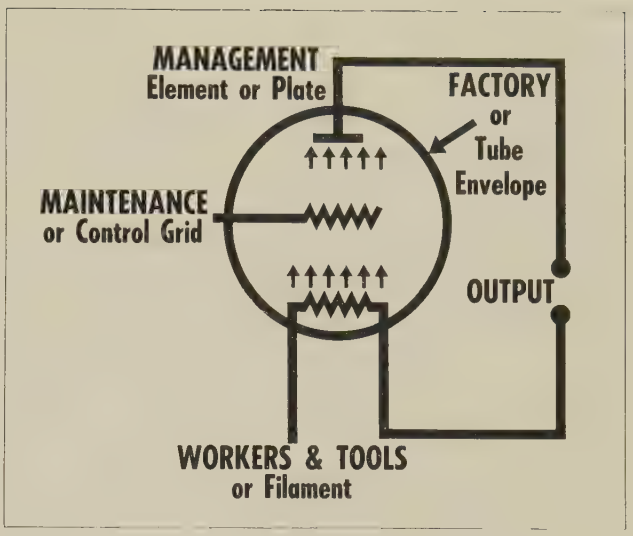
ization or valve, which, without further addition, can be made to produce varying qualities of goods.

The instant management starts to function we have applied a voltage or pressure to our plate circuit which permits

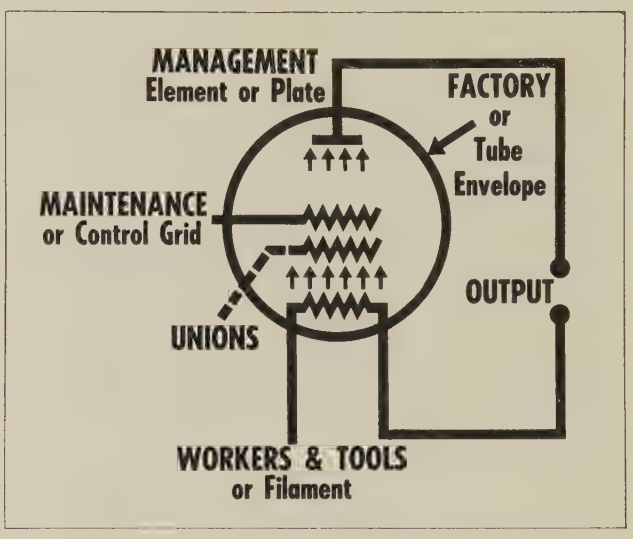


production to start by the emission of effort from the filament or worker and machine tool group. The amount of emission or work, and hence goods produced, will depend on the voltage or pressure from the plate or management element.

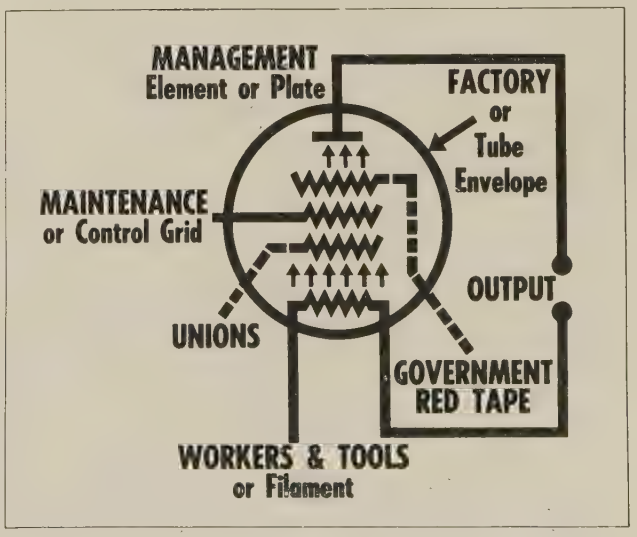




However, it is or should be clear that too much pressure from the plate circuit, or too much emission from the worker of filament, while possibly producing more goods, can very readily affect the quality of those goods.



It is at this point that we recognize the necessity for a third or controlling element for our tube. There are, of course, a number of operations that could affect the quality of a product but to me, and I am quite sure to you men, the most important



is maintenance of equipment. Let us assume that in our simple tube we add a third element which we will call the grid or maintenance control.

In the theory of the simple tube the filament or cathode is heated and starts to send out ions. However, without the guidance of the plate these ions can't go anywhere. We, therefore, apply a voltage or "know-how" to the plate and the ions, or in our case the goods, start flowing to the plate and out through the shipping channels. Due to a lack of maintenance the quality of these ions or goods is not all that it should be and the customers start to complain.

The smart company who realizes the importance of maintenance in the production of any item will add the third element or controlling grid to the valve and this he calls maintenance. By varying the voltage on the grid the flow of ions or goods can be controlled. In the layout of any manufacturing operation there must be a good balance between the plate voltage and the emission element or filament. Once this is established the amount and quality of the goods can and will, to a great extent, be determined by the voltage or maintenance impressed on the grid. In other words, gentlemen, maintenance, often considered as a stepchild by many who have not realized its importance, can be the difference between success and failure. I don't think that there is any group of maintenance men who know this fact better than you.

Next in importance to the establishment of a systematic preventive maintenance program with definite operations to be performed, is a record of what was found and what was done to correct the trouble and to prevent frequent reoccurrence. The very thought of the work involved in the keeping of records has a tendency to scare many away from the advantages to be gained. Actually, there is no necessity for a cumbersome system. A simple card record of each motor, generator, or control will, in most cases, suffice. However, the duty of keeping any record should be assigned to one person or it will fall by the wayside. The cost of one clerk for this job, among other details, will pay dividends.

Keeping of records is only part of the story. Unless we make a periodic analysis of what is occurring as shown by the records, and make the necessary changes which continuous failures indicate are advisable, we have gained little or no advantage from that part of our work.

CLEANING OF EQUIPMENT

Three of the greatest enemies of the continuous operation of electrical equipment are dirt, grease, and moisture. The first two of these elements can be found in almost any operation, but when they are mixed with atmospheres containing excessive moisture, the maintenance men really have something to contend with. Forgetting for the moment those climates where the atmosphere is inherently moisture laden, the papermaking industry has to operate continuously under the worst possible conditions.

This product starts out in life made up of approximately 97% water, all but about 5% of which must be driven off before the finished material is obtained. The result is that a lot of that water is thrown into the surrounding air. This moisture, by itself, is bad enough but when mixed with dirt and grease it makes a nice combination for the disintegration of insulation, the deterioration of lubricating oils and greases, and clogging up of ventilation ducts.

If you can't keep it dry at least keep it clean! This is one of the most important considerations of any maintenance program, the neglect of which can cause more grief than occasional overloading. Over the years the recommendations covering this operation have been pretty much the same involving the following simple instructions:

1. Wiping off direct with clean dry cloth. Note: *Do not use waste.*
2. Blowing out dirt with compressed air. Note: *Air pressure should not be over 50 pounds.*
3. Drawing dirt out of ducts with vacuum. (Dry conditions.)
4. Removing dirt with solvents.
5. Under extreme conditions washing off with water.

In general these instructions are so simple that they are too often neglected. In making use of them there are certain precautions to be taken. For example: when wiping be sure to wipe the dirt off and not into the machine. If the air pressure is too high in blowing operations damage to the insulation may result.

Solvents normally recommended are: petroleum distillates with flash point above 100°F., supplied by practically all oil companies. This list includes Stoddard solvent, mineral spirits, cleaners, naphtha, and others with flash point above 100°F. All of these are flammable and care should be taken to see that there are no fire hazards in the cleaning vicinity.

At one time carbon tetrachloride was freely recommended for stubborn cases. Today the use of this material is frowned upon by all safety-first men, because of the dangerous toxic effect. There are certain distillates which use a percentage of carbon tetrachloride to increase the flash point. These also should be avoided but, if used, care should be taken to see that the space used is well ventilated.

A practice which has come into use during the past few years is that of cleaning with ground corn cobs and an air pressure system. One company, the Carnegie Steel Co. at Gary, Ind. uses no other method, at least for the moment. This method of cleaning has the advantage of employing a dry element making it unnecessary to dry the machine after cleaning. Picture if you will a large motor over which a tarpaulin has been thrown. Enters into this enclosure a maintenance man with goggles, and air pressure hose with gun, and a tank containing the ground corn cobs. When he feels that the machine is sufficiently clean he can reverse the air pressure and suck out all of the loose corn cob from under the protective covering and the machine is now ready for operation. Regardless of the method keep your electrical apparatus clean.

During the past few years a relatively new method of cleaning known as the vapor degreaser, has come into use. It works on the principle of creating a hot vapor from perchlorethylene or similar chemicals which, when coming in contact with the cooler part to be cleaned, condenses on that part and running off carries the dirt and grease with it. It is fine for metal parts but we do not recommend it for cleaning armatures or other electrical apparatus containing windings. It may lift the varnish and carry dirt into the cracks.

It is just as important to keep your control clean as it is the rotating apparatus. On relatively high-voltage circuits with periodic opening of the contacts under current, the contacts of the circuit opening device will clean themselves. On low-voltage control circuits, however, such is not normally the case and dirty or corroded contacts may cause interruptions. Keep the contacts of all control apparatus clean and free from corrosion with file or sand paper (never use emery cloth). Do not use oil on hinge joints unless specifically designed for lubrication. Most contactors today use open knife edge fulcrums on the armatures. Oil attracts dirt and this creates, in time, a gum which tends to slow up the operation. Be sure to wipe off all filings that might get into hinged joints or create shorts between terminals.

LUBRICATION

To the average layman the word "maintenance" brings to his mind a picture of a man with a grease gun and oil can. The reason for this is, of course, obvious when you consider that his nearest approach to the subject is his automobile. About all he hears from the radio and papers is, "Have your car greased every 500 miles and change the oil every 1000 miles." Lubrication is important and it is one item of maintenance that has received the close attention of the design engineers.

It was realized long ago that human nature was frail and

anything requiring close attention would at times be neglected with resultant failures. To overcome this defect many automatic oiling schemes were devised to eliminate trouble from dry and hence hot and burned bearings. However, even here the human element was involved because someone has to see that the automatic feature was in working order.

Since the ingress of dirt and the exit of oil were two major elements contributing to the failure of the bearing, the problem was to provide a sealed container for the sleeve bearing. As you now know, great progress was made with the sealed sleeve bearing and others. Periods between oiling became greater and bearing failures were greatly reduced.

With the introduction of antifriction bearings new problems of lubrication became evident. We had to learn our lesson the hard way that too much grease was just as bad as too little. Again, we found that grease which was suitable for one type of operation was entirely unsuited for others. One phase of the ball bearing problem that is still not too thoroughly understood is that pertaining to the use of the so-called free fit bearing. In the early days of the application of antifriction bearings we ran into trouble on relatively high-speed motors due to galling of the races and balls. We found that it was a question of clearance and the solution, a bearing somewhat different than the market standard. This led to the use of a free fit bearing which is the same as the standard, except for greater clearance. It should be apparent that in repairing a motor the type of clearance in the bearing is important. Since bearings produced today still carry only the standard number such as 410–4100, etc., depending on the manufacturer, the carton in which it is shipped must be checked for the degree of fit. Again, the shop repairing the motor, unless they have the background of the design, may have no way of knowing that a free fit bearing is required. The original manufacturer knows.

Probably one of the greatest advances in the problem of lubrication was the introduction of the prelubricated sealed ball bearing by Westinghouse. When put on the market just a few years ago it was not a catch phrase but a well-proved idea. Before adopting this bearing as standard, motors with this form of lubrication had been in trial service over 5 years. Many motors so equipped are approaching 10 years without regreasing. Can you think of any other element that has contributed so much to the lubrication problem?

MOTORS IN GENERAL

We have now lived so long with the electric motor that we are inclined to take it for granted and forget some of the fundamentals. Everything that moves will wear in spite of the precautions taken by design engineers to reduce the effects of that wear. It should be obvious, therefore, that we must still carry on our daily, weekly, and monthly inspections if we are to expect more or less trouble-free continuous operation. Most of these inspections in a well-organized plant can be limited to the three senses—look, feel, and listen.

Look—For any outward appearance of faulty operation.
Feel—For vibration, looseness, and heat.
Listen—For any exceptional noise, knocking, or unusual sounds.

Naturally it is expected that the recommendations of the manufacturer will be followed in checking those things that he knows from past experience, will tend to give trouble.

In a highly competitive economy such as we still have in the United States, every manufacturer of electrical equipment is constantly striving to improve the quality of his produce and is, at the same time, awake to the effect the lasting quality of that product has on the continuing acceptance of his goods. He has finally realized that a reduction in maintenance and its cost is just as important as performance of function. To this end the following have been developed:

Longer Life Coils. Practically all early day motors were wound with coils insulated with class A or organic materials. While for many years the average temperature rise over the country on run-of-mine motors was only 26°C., a change has occurred in recent years due to reduction in size of machines and closer application. Fortunately, however, engineering developments have kept pace with the need for conservation of materials and have produced new insulating mediums to take care of those cases where excessive temperatures are encountered.

While the so-called class B coil is not new, its construction was limited to the use of mica and asbestos tapes. While mica is still the accepted standard for slot insulation, the asbestos tape has been almost completely superseded by glass. Asbestos tapes were only partially inorganic due to the need of an organic warp thread to give it strength. Of course glass threads can now be used in place of cotton for that purpose, but the finished tape would still be too bulky for present-day machines.

The use of improved inorganic insulation by itself was, of course, only part of the story since the binding varnishes were good for a maximum of only 125°C. Research again came to the rescue with an inorganic varnish now known as silicone. This material together with the glass fiber and mica permit the construction of coil known as class H with an adopted temperature of 180°C. This is playing it safe as we know through many tests that it will stand 250°C. without too much deterioration. All of which is of course designed to reduce maintenance in the extreme cases.

For standard run-of-the-mill integral horsepower motors it has not been necessary to go the extremes of class B or class H windings, except for some very special applications. However, great improvement over the old class A cotton insulated windings has been made with resulting fewer rewinds.

Making Full Use of High-Temperature Insulation. While it is true that we are not called on very often to provide for extremely high temperatures in motor and generator coils, there are such cases and other parts of the motor must be equipped with details that will not be too greatly affected by those temperatures. For the bearings normal grease will not do. We have, however, a new silicone grease which will take care of the situation.

For exceptionally high-temperature conditions on d.c. motors special insulation may be required for the commutator and a higher melting point alloy than that obtained with solder must be used to connect the armature coils to the commutator necks. While we have the solution for most of these problems not all of them are as yet commercial but they do show the trend.

Bearings. While the old style sleeve bearing did its job if carefully watched, it was, due to its construction, subject to a loss of oil and dirt which crept through its open construction. The development of the sealed sleeve bearing greatly reduced maintenance costs for this item by preventing, to a great extent, loss of oil and preventing dirt from getting into the bearing. The latest step involving the prelubricated sealed antifriction bearing has practically eliminated maintenance at this point.

These and numerous details have all helped to ease the maintenance man's problems but it would be a mistake to assume that a systematic program of inspection and eventually replacement of parts has or will become unnecessary.

ELECTRONIC EQUIPMENT

Much has been written about the wonders and mysteries of electronics and I don't doubt but what this has had a lot to do with some of the confusion that exists today. I have already shown you, by analogy, just how simple the electronic tube is, and I also pointed out how it can become complicated by the addition of foreign elements. The most important consideration in the maintenance of electronic control is a recognition of the fact that the approach is entirely different to the normal procedure for motors and the previously accepted controls.

In dealing with electronics we must remember that we have a new device which is a radical departure from the previously accepted mechanical devices. However, it is not a theoretical proposition but one that is here to stay and grow into one of our most useful tools. We already know that with increased paper speeds something beyond the accepted practice is necessary. Naturally there are and no doubt will be "bugs" to be ironed out but that is a natural sequence of all new developments. As time goes on it will be accepted as the only method when quick response is required and where the wear and tear of mechanical devices will tend to prohibit their use.

While it is true that there were numerous troubles with some of the early installations, it is equally true that others have performed with little or no trouble. Again, where troubles have occurred they have, to a great extent, been ironed out and the improved over-all performance has made the effort well worth while. The result has been that the trend is in the direction of a greater use of such devices.

Now as to the maintenance of these electronic devices, the best advice I have been able to obtain is to first see that the installation is in the cleanest possible location free from dirt and moisture. Second, watch the result of its operation in machine performance, and finally, leave them alone so long as the second qualification is met.

To help meet the second part of the above advice paper mill electronic control is equipped with meters to check performance. A reading of the meter by itself is only part of the story, but when checked against performance of the controlled machine, does give an indication of the correctness of operation. A check of day-by-day readings will show when variations occur and a necessity for replacing of the electronic unit at the earliest possible time.

I have been told that "down time" is worth about $1000 an hour to the producers of paper and anything that will help to reduce outages is like money in the bank. No doubt all of you have driven an automobile, and as with the operation of paper machines, you instinctively know that your engine is not acting quite right due possibly to a faulty plug. The car will run and no doubt get you to where you are going but at the earliest possible moment you will have it checked.

If you are mechanically inclined you may have extra plugs with you and at the first stop you will replace the faulty plug. So it is with your paper machine, except that in place of the spark plug, you will have a spare electronic unit. The faulty unit can then be returned to your maintenance shop for replacement of tubes and any other changes, by personnel trained for that purpose.

In closing I would like to reiterate the statement "When it is working within reasonable limits, leave it alone."

ADDENDA

Since there are a mass of detail operations involved in good maintenance practice and time will not permit a satisfactory coverage, the following list of documents will provide the necessary information:

1. Murphy, Louis J., Getting the most from electric drives in pulp and paper production. *Southern Pulp Paper Manuf.* (Feb. 15, 1951). *Note:* One of the most complete detail coverages of motor maintenance in paper mills.
2. Westinghouse, "Maintenance Hints," HB-6001F. *Note:* A general coverage of many types of electrical apparatus.
3. Sorokin, V. G., Vibrations in electrical machines. *Westinghouse Maintenance News*, Spring and Summer issue, 1947.
4. Baker, R. R., Electrical maintenance in paper mills in war time. Paper American Pulp & Paper Mill Supt. Assoc., Atlanta, Ga. (Nov., 1942).
5. Lynn, C., and Elsey, H. M., Effects of surface film on commutators. *Elec. Eng.* (June, 1949).
6. Elsey, H. M., Treatment of high altitude brushes. AIEE (May, 1945).
7. Brandt, E. C., Planned maintenance budget. Paper general—electrical superintendents, maintenance, and construction men.
8. Wartime electrical maintenance practices. *Electrical Contracting* (June, 1943).
9. Rylander, J. L., The effect of temperature on insulation resistance.
10. DeKiep, J., Hill, L. R., and Moses, G. L., The application of silicone resins to insulation for electric machinery. AIEE (Jan., 1945).
11. Ferris, R. E., and Moses, G. L., Fibrous glass for electrical insulation. *Elec. Eng.* (Dec., 1938).

Presented at the Engineering Conference of the Technical Association of the Pulp and Paper Industry, Savannah, Ga., Oct., 1951.

EMPLOYMENT SERVICE

Positions Open

P182-52. Young Chemical Engineer needed for liaison between sales and production departments on new material being offered papermaking industry. College graduate with some knowledge of paper manufacturing plus general interest in selling required. Excellent opportunity with young and growing company having wide range of products for defense and peacetime markets. Work leads to managerial position.

P183-52. Pulp and paper mill in North Carolina has opening for a Graduate Engineer, Mechanical, Civil, or Electrical in Plant Engineering Department. Recent graduate with up to five years' experience in industry preferred.

P184-52. Chief Chemist for an insulating board mill on the West Coast. Must have knowledge of coatings and wood chemistry.

P185-52. Chemical Engineer. For specialty mill in the Miami Valley. Prefer man with paper mill experience to conduct and supervise mill trials, routine and control tests. In replying state complete history and salary required.

P186-52. Chemist. Graduate or equivalent experience for development and control work in plastic coating division of an established paper manufacturer in the Miami Valley. Good starting salary—vacation with pay—hospital care, group insurance and other employee benefits.

P187-52. Rubber manufacturer requires paper chemist with good background in paper technology with the ability to initiate new latex developments in the paper field; also latex paint chemist for the development of latex paint formulas, to assist customers in the development and evaluation of rubber paint. Location—Connecticut. Please state age, experience, and educational background.

Positions Wanted

E152-52. Chemical Engineer. 20 years' technical and management experience, paper and cloth coating, coated abrasives, specialty paper products, coating and converting machinery, resins, and specialties. Desires position in research and development or engineering.

E153-52. Graduate Pulp and Paper Chemist. 15 years' diversified experience in research, product development, and technical control. Desires position as technical assistant to manager or superintendent of a progressive board mill. Presently employed.

E154-52. Technical graduate with extensive experience in papermaking and management desires position as assistant general superintendent with dependable and progressive paper manufacturer.

E155-52. Chemist. 20 years' experience in paper and paper coating; also supervision of stock refining; rope, jute and rag refining, deinking; white water control; and chlorination. Thorough knowledge of fourdrinier, cylinder, and Yankee machines, as well as off machine and on machine coating equipment, coating formulation and preparation.

E156-52. Engineer. Graduate civil and mechanical engineer, active and reliable, having had 30 years' extensive experience in design and manufacture of kraft, sulphite, groundwood, and tissue grades desires to make a connection in an operating or administrative capacity.

E157-52. Paper Chemist. B.A. in Pulp and Paper Technology June 1952. B.S. in chemistry 1949. More than 2 years' experience as chemist in pulp and paper mill. Married; age 26; Veteran. Desire position in production or development on West Coast.

E158-52. Director of package research, development, and merchandising now serving a leading manufacturer of packaging materials desires to administer a similar program for a consumer of packaging.

Instrumentation Studies. LXVII. Erasability of Paper

IV. A Proposed Method for Evaluating the Erasing Quality of Paper

W. A. WINK, N. J. BECKMAN, W. P. RANEY, and J. A. VAN DEN AKKER

WITHIN the past several years three reports have been issued on the subject of erasability (*1*). By way of brief review, the first of these covered a general discussion of the subject and presented definitions and statements obtained from experienced technical persons in the writing paper industry. The various factors that are called into play in judging the erasing quality of a paper were enumerated and consideration was given to the probable success or failure of adequately defining all the factors that are basic to the problem, with sufficient specificity to be really useful in a scientific measurement.

Parts II and III described the investigational phases of this study in which pertinent factors of the problem were investigated. The study was of a fundamental nature, conducted with research equipment; it was undertaken with the hope and plan that the information obtained would lead to improved definitions of the factors that are basic to erasability and also to instrumentation suitable for the laboratory evaluation of this property. Although adequately concise definitions of all of the pertinent factors have not yet been evolved, the information obtained throughout the course of this study has, nevertheless, given us a better understanding of several of the fundamental aspects of erasure, and it has aided us in the development of techniques which will provide a more objective means for evaluating the erasing quality of a paper.

The purpose of this report is to present a procedure for the evaluation of erasability, evolved from the studies that have been carried out under this program. In this procedure emphasis is placed on three general aspects of the problem—namely, (1) the ease of erasure, (2) the appearance of the erased surface and that of the rewritten characters, and (3) the function of the paper.

Of the properties to be evaluated, the ease of erasure, is most easily measured. The number of abrading strokes required to expunge written characters from the surface of the paper is used as a criterion for judging this property. By controlling such variables as the uniformity of the erasing wheel, the vertical thrust of the wheel on the paper, the relative humidity, and the method of application of the written characters, it is believed that reliable and reproducible results will be obtained.

The factors which come into play in judging the appearance of the erased surface and that of the rewritten characters are the most difficult to evaluate in a purely objective manner. Optical measurements are employed as a means for evaluating certain changes in the appearance which are perceptible to the eye but these measurements alone are not capable of detecting such changes as the removal of cockle or surface markings on the paper. The instrumental results corresponding to certain changes in the appearance of the surface of the paper must be supplemented by visual evaluations.

Evaluation of the writing quality of the erased surface is judged visually, preferably with the aid of pictorial standards. Lines are reproducibly drawn over the erased surface with a Schur-Levy rotary pen and then compared directly with a standard set of lines displaying various kinds of writing quality. Standards of this type have not yet been developed for general use; for the time being, it will be necessary for each laboratory group to prepare a set of their own standards.

The function of the paper is an aspect of erasability that relates to the specific properties that are desired in the erasing quality of a paper. The relative influence of penciled and typed characters on erasability data has not been studied thus far and conclusive statements cannot be made at this time. Since these studies exclusively involved the erasure of ink-drawn characters, the procedure presented in the following section is limited primarily to the erasure of inked characters.

TEST METHOD

1. *Abrader.* In our study of the various phenomena of erasability, all erasures were made with an abrader designed specifically for use as a research tool. Because of the desired flexibility of operation, this abrader by necessity embodied features which could conceivably be eliminated in an abrader intended primarily for use in the routine evaluation of erasability. For general use, a simplified model of the abrader described in Part II may be utilized; however, it is believed that such an abrader should embody the following features. The abrader shall be of the reciprocating type, having a motor-driven mechanism which will cause a circular erasing wheel to move back and forth over the surface of the paper at a speed of not more than 70 erasing strokes per minute. Stroke speeds in excess of this value may significantly affect the moisture content of the paper because of heating and, consequently, also affect the erasability data. The forward motion of the abrading wheel shall be the erasing stroke and shall be of nearly constant speed throughout its entire distance of travel. The assembly supporting the erasing wheel shall be equipped with a continuous type of ratchet which will lock the abrading

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wheel to prevent rotation on the forward erasing stroke and release to allow free rotation on the backward nonerasing stroke. Translation of the reciprocating motion of the motor-driven mechanism to the abrading wheel shall be accomplished by means of a simple clevis. Since the vertical thrust of the erasing wheel on the sample, during an erasing stroke, is affected by the vertical position of the bearing connecting the clevis to the driving mechanism, the position of this bearing shall be adjusted to lie in the horizontal plane containing the specimen. Means shall also be provided for adjusting the vertical thrust of the abrading wheel on the specimen to a value of 550 grams, when a wheel 0.45 inch in width is employed.

In order to erase a surface of a sufficient area for the subsequent optical measurements, the abrader shall be equipped with an automatic cross-feed mechanism. This mechanism shall be synchronized with the nonerasing stroke and shall cause the specimen to move in a direction perpendicular to that of the erasing wheel in such a manner than successive erasing strokes are made on fresh areas of the paper. To obtain uniformly abraded surfaces, it is important that the stepwise displacement of the specimen per stroke be an integral fractional multiple of the width of the erasing wheel. Accordingly, the displacement of the specimen per stroke shall be equal to one third of the width of the erasing wheel so that, upon the completion of three complete abrading strokes, the specimen is displaced a distance equal to the width of the wheel. The length of the stroke and the lateral travel of the specimen shall be adjusted to abrade a surface no less than 2 by 2 inches in size.

The surfaces of the specimen and the abrading wheel shall be kept clean and free of debris during the erasing process. To accomplish this, a fan and several small brushes shall be used. Small camel's hair brushes shall be mounted immediately before and after the erasing wheel so that, by their sweeping action, they dislodge loose fibers and debris from the surface of the specimen. A fine wire brush of the suede shoe brush variety shall be mounted against the abrading wheel for a similar purpose. A stream of air, obtained from a fan, shall be directed over the surface of the specimen to carry away the loosened debris. In general, it will be found that office fans are not satisfactory for this purpose. The air velocity produced by such a fan is too low to remove the heavy particles produced in abrasion, whereas blowers of the type found in vacuum cleaners have a tendency to raise the temperature of the air stream beyond tolerable limits. A fan having the desired characteristics for this application is one which may be constructed by mounting an 8-inch low-pitch model airplane propeller to a small vacuum cleaner motor. Air velocities in the neighborhood of 2800 f.p.m. are required to remove loose fibers and debris from those papers on which an appreciable amount of surface fuzz is developed. For papers which tend to become glossy during abrasion, air velocities somewhat less than the cited value are sufficient to keep the surface of the specimen clean.

"Calibrase" abrasion wheels, No. CS17F, manufactured by the Taber Instrument Co., shall be used as the abrading media. The investigation of the abrading characteristics of 21 different abrading wheels (all type CS17F), as described in Part III of this report, indicated that the variability of the abrasiveness of the wheels was of the same order of magnitude of, or possibly smaller than, the variability of the paper employed in the study. If the control over the manufacture of this type of wheel is such that the characteristics of their abrading action fall within the limits exhibited by the 21 wheels investigated, it is felt that the reproducibility of the resulting abrasions will be satisfactory.

2. *A reflectometer* suitable for evaluating the reflectivities R_∞ (reflectance of a specimen backed by a thick pad consisting of the same paper as that of the specimen) and R_0 (reflectance of specimen backed by a black body). Suggested instruments satisfactory for this purpose are the Bausch and Lomb opacimeter, the General Electric reflection meter (using the No. 6 filter), and the General Electric recording spectrophotometer.

3. *A special low-angle glossmeter.* In this instrument, the specimen is illuminated and viewed at an angle of 9° with the surface of the paper. Illumination is supplied by a 50-candle power single-filament lamp and the light reflected from the surface of the sample is measured by means of a Weston photronic cell and a suitable galvanometer circuit. The incident beam shall contain a rectangular aperture to limit the size of the illuminated spot on the specimen to a length of 1 inch and to a width of approximately $^1/_2$ inch. A similar aperture shall also be placed over the surface of the photocell and positioned to accept only the specularly reflected light. No lenses are employed in the interest of obtaining a small spread of angles in the incident beam. Accordingly, the intensity of the light reflected by a specimen is low, and a galvanometer of good sensitivity must be used. In standardizing the instrument and adjusting the "hundred setting" on the galvanometer, a piece of black Carrara glass is placed over the sample opening and a neutral filter is held in the path of the reflected beam. By standardizing in this manner, the galvanometer scale is spread by a known factor of approximately 10, since the neutral filter is removed while measuring the gloss of the paper. For those who do not wish to go through the trouble of setting up the galvanometer circuit described above, it will be of interest to know that the Welch Densichron, manufactured by the Welch Scientific Co., has adequate sensitivity for this special application.

4. *A special rotary pen,* developed by Schur and Levy, with facilities for drawing the pen over the paper at a uniform velocity.

The specimens shall be approximately 6 inches square and shall be free of abnormalities such as watermarks or obvious flaws.

The procedure employed for abrading the specimens and obtaining the desired physical data for evaluating erasability is as follows: The specimens for erasure are conditioned in a controlled atmosphere in accordance with TAPPI Standard T 402 m-49. After conditioning, reflectivity (R_∞ and R_0) and gloss measurements are made on the portion of the specimen to be abraded. The low-angle gloss measurements are taken for both the machine and cross-machine directions for each of the four 90° orientations of the paper. The average of the four readings obtained in this manner is

subsequently utilized for calculating the gloss ratio. After performing the optical measurements, a pair of ink lines, $^3/_4$ inch apart, are ruled on the specimen parallel to the machine direction of the paper for the specimens which are to be abraded with the erasing stroke parallel to the machine direction of the paper and perpendicular to the machine direction for those specimens which are to be abraded with the erasing stroke perpendicular to the machine direction of the paper. The lines are drawn with a Schur-Levy rotary pen employing an ink made in accordance with the formula given in TAPPI Standard T 431 m-45. The specimens, after application of the ink lines, are allowed to condition for a period of at least 10 minutes prior to performing the erasures.

Ten specimens are prepared in the foregoing manner and abraded, five with the abrading stroke parallel with the machine direction of the paper and five with the abrading stroke perpendicular to the machine direction of the paper. Each specimen is abraded for the number of strokes required to expunge the ink lines. The erasure is considered complete when the lines have been obliterated to the point where only a very small trace of the ink remains.

Following abrasion of the specimens, reflectivity (R_∞' and R_0') and low-angle gloss measurements are obtained on the portion of the specimen lying between the pair of obliterated ink lines. Because of the strong directional effects caused by the abrasion, it is important that the gloss readings again be obtained for the four 90° orientations of the paper; these, as before, are averaged and the ratio obtained by dividing the average of the "in" and "cross" direction readings after erasure by the average of the "in" and "cross" direction readings before erasure. Averages of the respective reflectivity values before and after abrasion are also obtained and the differences of the averages of the R_∞ values ($R_\infty - R_\infty'$) and the averages of the R_0 values ($R_0 - R_0'$) are calculated.

Upon completion of the optical measurements, a second pair of ink lines is drawn over the abraded surface adjacent to the original pair of lines. These are examined visually and compared with a set of pictorial standards for evaluating the writing quality of the erased surface. The lines are examined for continuity of the ink film, degree of feathering (if any), and for any other visible defects or imperfections.

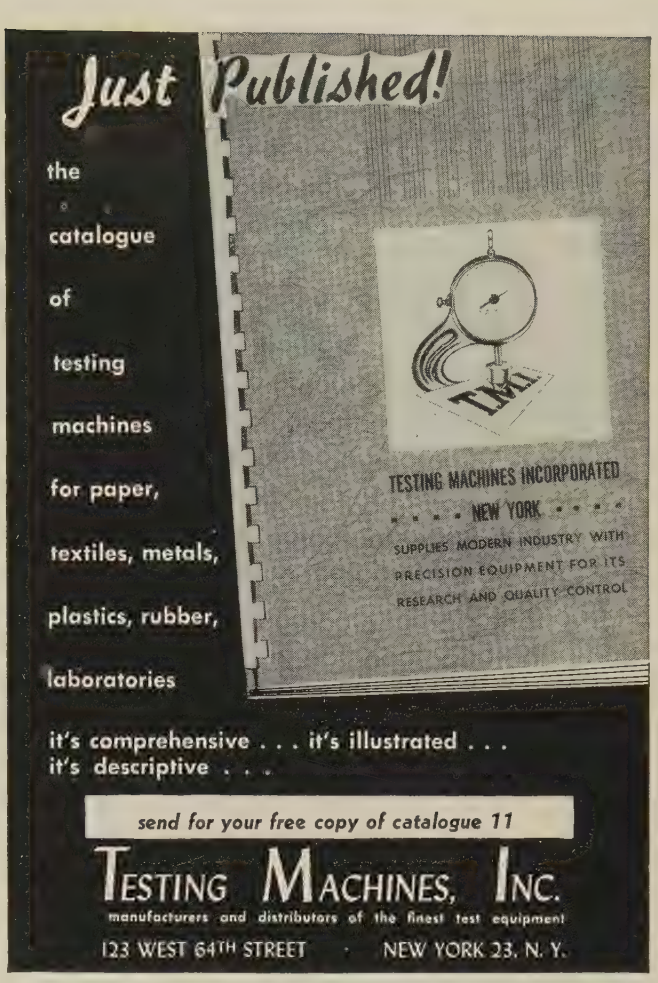
REPORT

The report shall include the following:

1. The number of abrading strokes over any given point of the abraded surfaces required to remove the ink lines. The number of abrading strokes is a basis for judging the ease of erasure.

2. The reflectivity R_∞ before and after abrasion and the difference between these two values. The difference in the reflectivity values will serve as an objective means for measuring the degree of discoloration caused be erasure. Differences in reflectivity of the order of 0.5 to 2.0% are distinguishable with the eye.

3. The reflectivity R_0 of the sample before and after abrasion and the difference between these two values. The change in the reflectance R_0 serves as a means for establishing the order of magnitude of the change in opacity caused by abrasion.



4. The ratio of the low-angle glossmeter readings obtained by dividing the average values (average of both machine and cross-machine direction readings) for the abraded portion of the sample by the average values (average of both machine and cross-machine direction readings) for the unabraded surfaces. A ratio greater than one indicates that the gloss on the abraded portion of the specimen is higher than that of the original specimen, whereas a ratio less than one indicates a decrease in gloss. These ratios are of significance in the evaluation of the degree of fuzziness or gloss developed during erasure.

5. A statement regarding observed changes in the surface characteristics of the paper—such as the removal of cockle. Changes of this sort which relate to the appearance are not detectable instrumentally.

6. A statement concerning the writing quality of the abraded and unabraded surfaces. Two important aspects related to judging the writing quality are the continuity or lack of continuity of the ink lines and the degree of feathering.

LITERATURE CITED

1. Report No. 43 to the American Paper and Pulp Association. Erasability of Paper. Part I. A general discussion of erasability (Feb. 15, 1948). *Tappi* 34, No. 2: 89 A–92 A (Feb., 1951). Part II. Progress report on a study of erasability (Feb. 15, 1948). *Tappi* 34, No. 3: 112 A–118 A (March, 1951). Part III. Progress report on a study of erasability (Feb. 1, 1949). *Tappi* 34, No. 7: 103 A–119 A (July, 1951).

Received Nov. 23, 1951. This report is one of a series issued as a part of the Instrumentation Program of the American Paper and Pulp Association. The data in this report were submitted to the Association Feb. 1, 1951. Part III of this study appeared in *Tappi* 34, No. 7: 103 A (1951).

UNITED STATES PATENTS ON PAPERMAKING

Second Quarter, 1951 — *Compiled by CLARENCE J. WEST*

THE following list of United States patents has been compiled from the current numbers of the Official Gazette of the United States Patent Office. Because, as a rule, only one claim is published in the Gazette, it is not claimed that the list is complete; also, it is possible that the list may contain some patents that do not apply specifically to pulp and paper manufacture.

Copies of any of the following patents can be obtained from the United States Patent Office, Washington 25, D. C., by sending twenty-five cents for each patent desired.

April 3, 1951

2,547,005. Container. John H. Herrick, Wellesley, Mass., and John P. Pancoast, Glen Rock, N. J. Filed March 31, 1949. 2 claims. (Cl. 229-90.) A corrugated liner is provided for the inwardly folded end of a container for lotions, drugs, and similar bottled goods.

2,547,097. Thermoplastic bag construction. Gilmore T. Schjeldahl and Donovan D. Wendt, assignors to Bemis Bro. Bag Co., Minneapolis, Minn. Filed Nov. 7, 1949. 1 claim. (Cl. 229-62.) A polyethylene food-storage bag can be heated sealed and reopened a number of times.

2,547,208. Method for the refining of tall oil soap. Torsten Hasselstrom and Michael Stoll, assignors to U. S. Industrial Chemicals, Inc., Newark, N. J. Filed June 26, 1947. 6 claims. (Cl. 260-97.7.) A crude tall oil soap is freed from nonsaponifiable components and color bodies by extracting with boiling methyl isobutyl ketone.

2,547,362. Combined container and straw. Roger W. Berry, Princeton, Ill. Filed July 3, 1948. 6 claims. (Cl. 229-7.) A waxed paper beverage container incorporates a pair of drinking straws.

2,547,371. Electrolytic condenser. Joseph B. Brennan, Bratenahl, Ohio, assignor to Everett D. McCurdy, trustee. Filed Sept. 18, 1947. 3 claims. (Cl. 175-315.) Porous, soft paper, less than 0.003 inch thick, is treated on one side with aluminum particles.

2,547,395. Container. George D. Jansen, assignor to Jansen Container Co., Chicago, Ill. Filed Dec. 31, 1948. 3 claims. (Cl. 229-5.5.) A lightweight ice cream container with a flanged bottom.

2,547,429. Isolation of enzymes from aqueous solutions with lignin and gelatin and product obtained thereby. Ralph T. Alba, assignor to Wallerstein Co., Inc., New York, N. Y. Filed Oct. 26, 1945. 14 claims. (Cl. 195-64.) Enzymes (e.g., malt diastase) can be removed from aqueous solution or suspension as an enzyme-lignin-gelatin complex; alkali lignin is preferably employed.

2,547,628. Folding Carton. Ransom C. Ellsworth, assignor to The Ohio Boxboard Co., Rittman, Ohio. Filed June 2, 1947. 2 claims. (Cl. 229-33.) A tray-type folding carton embodies interlocking side and end wall flanges.

2,547,671. Fire-resistant article, method of making, and impregnant therefor. Morris J. Sostmann and Irwin L. Phillips, assignors to Philadelphia Textile Finishers, Inc., Norristown, Pa. Filed Oct. 13, 1945. 3 claims. (Cl. 252-8.1.) One part of antimony trioxide and two parts of acetic anhydride are boiled until solution results and mixed with chlorinated paraffin.

2,547,716. Foldable vending tray for ice-cream cups and the like. Elsie M. Murphy, Elmhurst, Ill. Filed Jan. 4, 1949. 3 claims. (Cl. 224-45.) A foldable portable chipboard tray for ice cream cups.

2,547,830. Treating and refining machine for pulp materials Charles W. Morden, assignor to Morden Machines Co., Portland, Ore. Filed Jan. 11, 1947. 6 claims. (Cl. 92-27.) The refiner consists of a conically shaped rotor with an exterior attritioning surface and a nonrotating shell with an interior attritioning surface surrounding the rotor, a receiving chamber at the small end of the shell, and a semifloating mounting for the shell.

2,547,849. Box construction. William F. Barry, Los Angeles, Calif. Filed Oct. 25, 1946. 11 claims. (Cl. 229-23.) A multiple-wall, reinforced cardboard box is adapted to support its contents at a distance from the top, bottom, and side walls.

2,547,871. Closure for paper containers. Douglas Kirk, assignor to The Quaker Oats Co., Chicago, Ill. Filed July 8 1947. 1 claim. (Cl. 229-43.) A die-formed, removable, and replaceable cardboard closure is adapted for a cylindrical dispensing container.

2,547,892. Container for paper napkins. Robert B. Stevens, Greenwich, N. Y. Filed Oct. 7, 1947. 4 claims. (Cl. 206-57.) A paperboard container for napkins has removable top.

2,547,899. Pressure mechanism for box forming machines. Julius A. Zinn, Jr. Chicago, Ill. Filed June 4, 1947. 22 claims. (Cl. 93-44.1.) A high-speed, fluid-operated, and fluid-controlled carton-forming machine.

2,547,913. Process for the preparation of vanillin and vanillin derivatives. Paul Lagally, assignor to Aschaffenburger Zellstoffwerke, Aktiengesellschaft, Redenfelden/Oberbayern, Germany. Filed Feb. 13, 1950. 5 claims. (Cl. 260-600.) Lignin (e.g., spent sulphite liquor) is heated to a temperature above 100°C. in an alkaline medium with a water-soluble, oxygen-liberating sulphoxide, tertiary amine oxide, or an iodoso compound to yield vanillin and vanillin derivatives.

April 10, 1951

2,547,948. Process for forming magnetic record members from a papermaking fiber slurry. Otto Kornei, assignor to The Brush Development Co., Cleveland, Ohio. Filed July 21, 1947. 3 claims. (Cl. 92-21.) A paper tape for magnetic tape recorders is made from pulp containing permanently magnetizable iron oxide.

2,547,963. Box flap gluing device. Morris P. Neal, assignor to A-B-C Packaging Machine Corp., Quincy, Ill. Filed Oct. 15, 1945. 16 claims. (Cl. 91-51.) The apparatus can be inserted into and removed from a carton-forming machine when it is desired to convert from one box size to another.

2,547,997. Molding from fiber dispersions. Harold E. Bowers, assignor to The New England Box Co., Greenfield, Mass. Filed March 19, 1946. 7 claims. (Cl. 92-55.) Liquid suspensions of fibrous materials (e.g., mechanical pulp, flax straw, etc.) containing naphthalene or camphor are molded and the filler is removed after drying the molded article; products of predetermined apparent density can be prepared.

2,548,001. Display shipping container. Sydney T. Butterfill, Hamilton, Ontario, Canada. Filed Jan. 9, 1948. 1 claim. (Cl. 206-45.21.) A multicompartment paperboard container for fresh fruit has small ventilating holes in the side walls and lid.

2,548,108. Flow distributor. Lloyd Hornbostel, assignor to Beloit Iron Works, Beloit, Wis. Filed May 31, 1945. 10 claims. (Cl. 92-44.) A headbox is equipped with an adjustable baffle and one or more imperforate rolls; the combination is used to control the head of stock flowing onto the wire.

2,548,130. Container. Elroy R. Stephan, assignor to Downing Box Co., Milwaukee, Wis. Filed Nov. 4, 1948. 11 claims (Cl. 229-23.) A fiberboard beverage case.

2,548,251. Means and method for securing the effects of end dipping in gas-tight flexible wall packages. Samuel Bergstein, Cincinnati, Ohio. Filed April 10, 1946. 21 claims. (Cl. 229-5.5.) Method and means for molding a thermoplastic, gas-tight coating substance about the ends of a flexible paper container.

2,548,252. Box with attached handles. Samuel Bergstein, Cincinnati, Ohio. Filed Aug. 3, 1946. 7 claims. (Cl. 229-52.) A one-piece cardboard garment box has a tear-resistant, solid kraft board carrying handle.

2,548,364. Pressure-sensitive record materials. Barrett K. Green and Robert W. Sandberg, assignors to The National Cash Register Co., Dayton, Ohio. Filed July 13, 1948. 28 claims. (Cl. 282-28.) A record material is highly sensitive to impact pressure such as typewriter or letterpress work. Attapulgite may be the solid color-forming substance and crystal violet or malachite green lactone the liquid.

2,548,365. Process for making pressure-sensitive record materials. Barrett K. Green and Robert W. Sandberg, assignors to The National Cash Register Co., Dayton, Ohio. Filed July 13, 1948. 47 claims. (Cl. 117-36.) A pressure-sensitive record

material is claimed on which a color mark can be produced by a localized pressure.

2,548,366. Manifold record material and process for making it. Barrett K. Green and Robert W. Sandberg, assignors to The National Cash Register Co., Dayton, Ohio. Filed July 31, 1948. 27 claims. (Cl. 282-28.) A manifold record material, which is smudgeproof and white on both sides, is coated with a color reactant material.

2,548,370. Box or carton stripper machine. Eric L. Hedstrom, Martin C. Otto, and Cecil E. Gooderham, Buffalo, N. Y. Filed Nov. 18, 1946. 8 claims. (Cl. 93-36.) The apparatus is adapted for the stripping of waste from the die-cut portions of paper stock after passage through a cutting and creasing machine.

2,548,417. Apparatus for thickening liquid slurry stock. Joseph Baxter, Jr., assignor to The Black-Clawson Co., Hamilton, Ohio. Filed Dec. 24, 1948. 12 claims. (Cl. 210-63.) A centrifugal thickener.

2,548,425. Beater. George W. Dodge, assignor to The Noble & Wood Machine Co., Hoosick Falls, N. Y. Filed Jan. 15, 1947. 3 claims. (Cl. 92-22.) The bedplates are contained in a solid frame, beneath which is located a series of hydraulic cylinders; the cylinder pistons serve to move the bedplate combination upwardly toward the stationary beater roll.

2,548,451. Apparatus for forming paper strips into continuous lable forming bands for yarn and the like. Stanley E. Tribbey, assignor to Caron Spinning Co., Rochelle, Ill. Filed March 29, 1949. 5 claims. (Cl. 93-81.) A machine for the high-speed, automatic production of paper bands for the banding of skeins of yarn.

2,548,513. Method of making high wet strength paper. Fred W. Boughton, assignor to Eastman Kodak Co., Rochester, N. Y. Filed April 22, 1944. 5 claims. (Cl. 92-21.) Pulp containing precipitated material is treated with melamine-formaldehyde or melamine-urea-formaldehyde resin; the resulting paper does not cause deterioration of light-sensitive chemicals of an emulsion coating.

2,548,520. Copolymers of proteins having unsaturated radicals united therewith. Rudolph E. Damschroder and John W. Gates, Jr., assignors to Eastman Kodak Co., Rochester, N. Y. Filed Feb. 13, 1947. 8 claims. (Cl. 260-8.) The products are suitable as paper sizing.

2,548,636. Stabilized cuprammonium hydroxide solution and method of preparing the same. Clark E. Thorp, assignor to Hawley Products Co., St. Charles, Ill. Filed May 29, 1946. 11 claims. (Cl. 23-190.) The cuprammonium hydroxide is stabilized by the addition of 0.3–2% by weight of hydroquinone, resorcinol, *p*-methylaminophenol, or ethylenediamine.

2,548,646. Moldproofing mineral coated papers. John A. Bicknell and Stanely J. Johnson, assignors to S. D. Warren Co., Boston, Mass. Filed Jan. 2, 1946. 3 claims. (Cl. 106-15.) Casein in an aqueous solution is mixed with the sodium salt of pentachlorophenol and sodium pyrophosphate.

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2,548,872. Method of fixing particles to sheet material. Bert S. Cross and George P. Netherly, assignors to Minnesota Mining and Manufacturing Co., St. Paul, Minn. Filed Aug. 10, 1948. 6 claims. (Cl. 117-17.) A particulate coated sheet article adapted to permit the transfer of its particles to a base sheet coated with a permanent binder.

2,548,942. Air filter. Raymond L. Brown, assignor to Electrolux Corp., Old Greenwich, Conn. Filed June 6, 1947. 2 claims. (Cl. 183-51.) An air filter for suction cleaners consists of a pad of felted combed cotton encircled by a ring of spirally wound kraft paper.

2,548,980. Pressure-sensitive adhesive tape. Waldo Kellgren, assignor to Minnesota Mining & Manufacturing Co., St. Paul, Minn. Filed Feb. 1, 1946. 1 claim. (Cl. 206-59.) The tape has a creped paper backing.

2,548,985. Skeleton carton. Stephen Lighter, Whitefish Bay, Wis. Filed Nov. 8, 1948. 17 claims. (Cl. 206-45.31.) A framelike cardboard container for display of relatively small articles.

2,549,000. Apparatus for applying adhesive to box blanks and the like. Charles E. Palmer, assignor to Package Machinery Co., East Longmeadow, Mass. Filed Oct. 9, 1948. 11 claims. (Cl. 91-47.) A machine for printing adhesive on a selected area on one side of a carton blank.

2,549,048. Liquid-tight carton and method. Frank D. Bergstein, Cincinnati, Ohio. Filed May 9, 1947. 18 claims. (Cl. 229-5.5.) Paperboard milk container with means for determination of the cream line.

2,549,138. Loud-speaker diaphragm. Clifford E. Stevens, assignor to Stevens Products, Inc., East Orange, N. J. Filed Oct. 4, 1946. 1 claim. (Cl. 181-32.) A molded pulp loud-speaker diaphragm.

2,549,139. Cone diaphragm for loud-speakers. Clifford E. Stevens, assignor to Stevens Products, Inc., East Orange, N. J. Filed June 17, 1947. 3 claims. (Cl. 181-32.) A molded pulp loud-speaker diaphragm has high sensitivity and a wide frequency-response range.

2,549,177. Sized paper, composition therefor, and method of making same. Paul B. Davidson, assignor to Strathmore Paper Co., Springfield, Mass. Filed March 21, 1949. 13 claims. (Cl. 117-156.) A blueprint paper with high wet strength is sized with a mixture of modified starch and glyoxal.

2,549,199. Combination envelope and letter paper. Nicholas Hodska, Stratford, Conn. Filed July 12, 1948. 5 claims. (Cl. 229-921.)

2,549,367. Method of recovering from pyrite the valuable constituents thereof. Charles W. Clark, assignor to Noranda Mines, Limited, Toronto, Canada. Filed Oct. 25, 1945. 5 claims. (Cl. 23-179.) Sulphur, sulphur dioxide, and iron oxide can be recovered on distillation.

2,549,373. Dispensing device for cartons. Dabney W. Forsyth, Santa Monica, Calif. Filed May 9, 1950. 4 claims. (Cl. 229-17.) A slide-type closure for a dispensing container.

2.549,568. Dispensing display carton. Hubert V. Bolding, assignor to The Bradley & Gilbert Co., Louisville, Ky. Filed July 19, 1948. 2 claims. (Cl. 206-44.11.) A carton for displaying pencils and a method of protection during shipment.

2,549,582. Opening means for regular cartons. Robert M. Dunning, assignor to Waldorf Paper Products Co., St. Paul, Minn. Filed Aug. 21, 1947. 8 claims. (Cl. 229-17.) A triangular tab is incorporated into the container.

2,549,682. Foldable box, Alice J. Grossniklaus, Wilmot, Ohio. Filed Aug. 15, 1946. 3 claims. (Cl. 229-16.) A folding paper box has a greaseproof liner which serves as a mold.

2,549,713. Method of preparing cellulose for subsequent treatment with sodium hydroxide. Joseph Seiberlich, assignor to Research Corp., New York, N. Y. Filed April 6, 1948. 9 claims. (Cl. 260-212.) Cellulose is treated with a chlorinated hydrocarbon containing not more than three carbon atoms.

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2,549,799. Collapsible cellular partition for containers. Walter C. George, assignor to Gaylord Container Corp., St. Louis, Mo. Filed Sept. 15, 1947. 7 claims. (Cl. 229-42.) Collapsible, cellular, strip-type partitions for bottle cartons are fastened by stitching or mechanical fasteners.

2,549,800. Collapsible container and cellular partition unit. Walter C. George, assignor to Gaylord Container Corp., St. Louis, Mo. Filed Oct. 3, 1947. 2 claims. (Cl. 229-42.) A folding, multicompartment, corrugated container is equipped with strip-type partitions which form cells for bottles.

2,549,801. Collapsible container partition and liner unit. Walter C. George, assignor to Gaylord Container Corp., St. Louis, Mo. Filed Oct. 6, 1947. 2 claims. (Cl. 229-42.) Collapsible, strip-type partitions for a bottle carton are provided with liner members for the side walls and/or bottom of the container.

2,549,802. Collapsible cellular container partition. Walter C. George and Arthur P. Klasing, assignors to Gaylor Container Corp., St. Louis, Mo. Filed Feb. 4, 1948. 3 claims. (Cl. 229-42.) The partition is designed to provide horizontal ventilation through the cells and to prevent the movement of the partition parts.

2,549,822. Binder for foundry compositions. Wilbur E. Koonce. assignor to Masonite Corp., Laurel, Miss. Filed Nov. 24,1947. 7 claims. (Cl. 22-188.) The binder is a hemicellulosic extract containing pentosans and hexosans 60–80 parts, lignin 5–25 parts, nonsugar carbohydrates 5–15 parts, and gums 2–8% parts.

2,549,849. Cork substitute and process for making same. Robert S. Owens, Charlottesville, Va. Filed Sept. 8, 1948. 11 claims. (Cl. 106-134.) A cork substitute consists of paper birch bark and a phenol-formaldehyde resin.

2,549,931. Bag or envelope. Irwin A. Reiss, assignor to Associated Paper Converting Corp., Los Angeles, Calif. Filed June 23, 1948. 1 claim. (Cl. 229-54.) An envelope-type paper bag for light merchandise.

2,549,966. Apparatus for the manufacture of metalized paper for electric condensers. Richard A. Grouse, assignor to A. H. Hunt Limited, Wandsworth, London, England. Filed June 26, 1946. 7 claims. (Cl. 219-19.) The apparatus is designed for the production of a paper-sheet dielectric which is partially metalized on one side.

2,549,970. Package. Stanley R. Howard, assignor to Pneumatic Scale Corp., Limited, Quincy, Mass. Filed March 27, 1946. 1 claim. (Cl. 229-14.) A cereal package consists of a waxed paper bag which is enclosed in a rectangular cardboard sleeve.

2,549,973. Paper shredding apparatus. Matthew J. Klein, Maywood, Ill. Filed April 19, 1950. 3 claims. (Cl. 164-84.5.) The shredder has two steel tearing rollers, one of which is jacketed in a rubber sleeve.

2,550,000. Bag structure and process of manufacture. Charles V. Brady and Russell J. Williams, assignors to Bemis Bro. Bag Co., St. Louis, Mo. Filed Oct. 15, 1945. 6 claims. (Cl. 229-57.) The patent covers a flat, pillow-shaped paper bag which may have various bottom forms.

2,550,143. Construction board and method of making same. Ernst Eger, assignor to United States Rubber Co., New York, N. Y. Filed Nov. 29, 1947. 14 claims. (Cl. 260-17.2.) A fibrous material is mixed with dispersed polychloroprene and a resorcinol-formaldehyde resin and dried.

2,550,192. Fourdrinier machine. Harry H. Hart, assignor to The Pusey & Jones Corp., Wilmington, Del. Filed June 9, 1947. 1 claim. (Cl. 92-44.) Screen-guiding and -tensioning means are designed to cooperate with a nonmetallic machine wire.

2,550,326. Planographic printing. Clarence A. Brown, assignor to Jones Graphic Products Co., Albuquerque, N. M. Filed Jan. 12, 1949. 12 claims. (Cl. 101-149.2.) A sheet of metal foil is laminated between two sheets of water-resistant paper; one outer surface is coated with water-soluble urea-formaldehyde resin, polyvinyl alcohol, and clay.

2,550,417. Novelty paperboard carton. Clarence F. Klein, assignor to Frankenberg Bros., Inc., Columbus, Ohio. Filed May 24, 1948. 5 claims. (Cl. 229-8.) A candy box with a cellophane window is suitable as a toy.

2,550,466. Manifold record material and process for making it. Barrett K. Green and Robert W. Sandberg, assignors to The National Cash Register Co., Dayton, Ohio. Filed Sept. 7, 1950. 2 claims. (Cl. 282-28.)

2,550,467. Manifold record material and process for making it. Barrett K. Green and Robert W. Sandberg, assignors to The National Cash Register Co., Dayton, Ohio. Filed Sept. 7, 1950. 2 claims. (Cl. 282-28.)

2,550,468. Manifold record material and process for making it. Barrett K. Green and Robert W. Sandberg, assignors to The National Cash Register Co., Dayton. Ohio. Filed Sept. 7, 1950. 2 claims. (Cl. 282-28.) In these patents, one surface of the sheet is coated with small solid particles of a color-reactant material (a zeolite) and the other with a pressure-rupturable material; when superimposed, pressure produces a color reaction.

2,550,469. Manifold record material and process for making it. Barrett K. Green and Robert W. Sandberg, assignors to The National Cash Register Co., Dayton, Ohio. Filed Sept. 7, 1950. 2 claims. (Cl. 282-28.)

2,550,470. Pressure-sensitive record material. Barrett K Green and Robert W. Sandberg, assignors to The National Cash Register Co., Dayton, Ohio. Filed Sept. 15, 1950. 2 claims. (Cl. 282-28.)

2,550,471. Pressure-sensitive record material. Barrett K. Green and Robert W. Sandberg, assignors to The National Cash Register Co., Dayton, Ohio. Filed Sept. 15, 1950. 2 claims. (Cl. 282-28.)

2,550,472. Pressure-sensitive record material. Barrett K. Green and Robert W. Sandberg, assignors to The National Cash Register Co., Dayton, Ohio. Filed Sept. 15, 1950. 2 claims. (Cl. 282-28.)

2,550,473. Pressure-sensitive record material. Barrett K. Green and Robert W. Sandberg, assignors to The National Cash Register Co., Dayton, Ohio. Filed Sept. 15, 1950. 2 claims. (Cl. 282-28.) In these patents a pressure-senstive record material incorporates two color reactants, one solid and one liquid, which are arranged in separate overlying layers on a paper web, so that marking pressures applied vertically to the layers will cause a penetration of the color-reactant substances of one layer into that of the other layer.

2,550,516. Mechanism for sealing cartons. Leonard Back, assignor to The Interstate Folding Box Co , Middletown, Ohio. Filed April 27, 1946. 21 claims. (Cl. 93-49.) The mechanism produces a tight, siftproof closure on seal end-type paper cartons.

2,550,520. Joint structure for laminates. Clarence G. Bennett, San Mateo, Calif. Filed Nov. 12, 1947. 3 claims. (Cl. 229-48.) A joint for a cellophane-vegetable parchment blank is described.

2,550,552. Stock inlet. John E. Goodwillie, assignor to Beloit Iron Works, Beloit, Wis. Filed Sept. 5, 1946. 2 claims. (Cl. 92-44.) A stock chamber is provided with rectifier rolls and baffles; the stock is fed from the outlet at high velocity.

2,550,582. Interlocking corner construction for containers. Reginald B. Meller, Oakland, Calif., assignor to Bemiss-Jason Co. Filed Aug. 14, 1945. 7 claims. (Cl. 229-35.)

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2,550,676. Combined waste-heat boiler and chemical recovery unit. David Dalin, Sodertalje, Sweden. Filed Oct. 17 1945. 4 claims. (Cl. 23-262.)

2,550,691. Noncorrosive detergent compositions. Jay C. Harris, assignor to Monsanto Chemical Co., St. Louis, Mo. Filed Nov. 25, 1949. 7 claims. (Cl. 252-140.) One of the constituents is the condensation product of tall oil with ethylene oxide.

2,550,730. Diestock and process of making same. Charles F. Story, assignor to Masonite Corp., Laurel, Miss. Filed Jan. 20, 1947. 6 claims. (Cl. 154-116.) Dense, laminated slabs are formed from a number of relatively thin sheets of fibrous lignocellulosic material, preferably from hardwood. The edges of the slabs are sealed against the penetration of moisture.

2,550,774. Lump-breaker roll for papermaking machines. Everett W. Clem, assignor to Rice Barton Corp., Worchester Mass. Filed June 22, 1946. 7 claims. (Cl. 92-48.) A lump-breaker roll is adapted for use with the couch roll of a fourdrinier machine.

2,550,776. Papermaking machine flow box. Everett W. Clem, assignor to Rice Barton Corp., Worcester, Mass. Filed March 3, 1947. 1 claim. (Cl. 92-44.) Compression waves are passed through the pulp slurry as it travels through the headbox.

2,550,920. Wrapping machinery. Joseph A. Gilbert, assignor to Rose Brothers (Gainsborough) Limited, Gainsborough, England. Filed Oct. 3, 1946. 4 claims. (Cl. 93-2.) A method for wrapping a bar of soap or a package of cigarettes in a paper, film, or foil wrapper.

2,550,928. Article controlled adjustment for packaging apparatus. James A. Kay, assignor to Rose Brothers (Gainsborough) Limited, Gainsborough, England. Filed Oct. 5, 1944. 15 claims. (Cl. 93-6.) The apparatus is designed for the closing and sealing of containers.

2,551,087. Polyethylene-wax laminating composition. Philip S. Barnhart and John C. Miller, assignors to Westfield River Paper Co., Inc., Russell, Mass. Filed Nov. 3, 1945. 7 claims. (Cl. 260-27.) A paper-laminating cement comprises 75% microcrystalline wax, 24% polymerized rosin, and 0.1% polyethylene.

2,551,164. Reinforced knockdown box. William A. Ringler, assignor to The Gardner Board and Carton Co., Middletown, Ohio. Filed Sept. 6, 1946. 1 claim. (Cl. 229-23.) A carton adapted for shipment and storage of folded blankets and the like.

2,551,186. Carton forming machine. Louis Veyret and Georges Geffroy, assignors to La Cellophane, Paris, France. Filed Dec. 31, 1947. 2 claims. (Cl. 93-51.) A carton blank is so folded that, in the folded carton, no open seams which require sealing extend below the level to which the carton is to be filled.

2,551,324. Container. Robert T. Frieders and John P. Strange, Jr., assignors to John Strange Pail Co., Menasha, Wis. Filed April 7, 1947. 4 claims. (Cl. 229-36.) A paperboard frozen food container.

2,551,335. Web cutting mechanism. Thomas E. Piazze, assignor to Shellmar Products Corp., Mount Vernon, Ohio. Filed March 29, 1946. 9 claims. (Cl. 164-66.) A rotatable knife and an oscillatable anvil.

2,551,450. Container. Leslie A. Minton, Portland, Ore. Filed Dec. 3, 1947. 2 claims. (Cl. 229-15.) A paperboard milk container.

2,551,527. Apparatus for closing and sealing containers. Robert B. Cartwright, Lincoln, Neb., and William M. Owen, Peoria, Ill.; said Owen assignor to said Cartwright. Filed May 26, 1948. 12 claims. (Cl. 93-2.) Method of sealing the flaps of a paper cereal container.

2,551,559. Bottle carrier. Clyde L. Gilbert, assignor to Owens-Illinois Glass Co., Toledo, Ohio. Filed March 20, 1948. 2 claims. (Cl. 229-28.) The carrier has an integral latch lock which locks the carrier in its expanded form.

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2,551,679. Divisible carton. Earl F. Johnson, Milwaukee, Wis. Filed Feb. 27, 1947. 8 claims. (Cl. 229-15.) A carton for canned and bottled goods may be divided with a knife to form two complete half-carton containers.

2,551,814. Container. Fred L. Rushing and Benjamin N. Williams, assignors to Gaylord Container Corp., St. Louis, Mo. Filed Dec. 13, 1945. 3 claims. (Cl. 229-45.) A corrugated shipping carton includes releasable means for locking a slip cover to the body of the carton adjacent to its upper corners.

2,551,854. Egg carton. Louis Schwartzberg, Chicago, Ill. Filed Aug. 10, 1948. 2 claims. (Cl. 229-29.) A paperboard egg carton incorporates a cushioning construction between transversely adjoining egg-receiving cavities.

2,551,946. Machine for working paper stock and other materials. Anton J. Haug, Nashua, N. H. Filed Aug. 21, 1947. 5 claims. (Cl. 92-26.) A refiner for pulp mill rejects.

2,552,051. Paper sundae dish. Walter P. Margulies, assignor to Lily-Tulip Cup Corp., New York, N. Y. Filed June 7, 1949. 2 claims. (Cl. 229-1.5.) The dish incorporates a frustoconical side wall which merges at its smaller end in a reverse conical hump.

2,552,127. Paper carton machine. Charles T. Walter, Lowell R. Newton, and Heinrich E. Haase, assignors to Swift & Co., Chicago, Ill. Filed March 23, 1943. 15 claims. (Cl. 93-55.1.) A machine is adapted for applying a skirted paper cap to a cylindrical container.

2,552,189. Paper weight indicating equipment. Hans P. Kuehni, assignor to General Electric Co., Schenectady, N. Y. Filed April 27, 1946. 3 claims. (Cl. 73-144.) A method for measuring the weight and/or tension of a traveling paper web.

2,552,340. Merchandise container having opening means. George A. Moore, assignor to Reynolds Metals Co., Richmond, Va. Filed June 12, 1946. 2 claims. (Cl. 229-51.) A container for candy bars, cigarettes, and the like.

2,552,474. Conical paper dish and blank therefor. Walter E. Amberg, assignor to Lily-Tulip Cup Corp., New York, N. Y. Filed Oct. 29, 1947. 2 claims. (Cl. 229-1.5.) A conical paper dish has flaps to engage fingers in the bowl of a paper-dish holder.

May 15, 1951

2,552,597. Process of making a molded article from lignocellulose. Warren R. Smith, Edmund R. Mellenger, and Donald F. Othmer, assignors to Donald F. Othmer, William R. White, Jr., and Louis Ricciardi, Coudersport, Pa. Filed July 9, 1947. 3 claims. (Cl. 106-163.) A wallboard is made from wood waste and sulphur by pressure and heat.

2,552,612. Continuous inpregnation of formed fibrous material. Douglas D. Adams, Karl Rollius, and John D. Risher, Canton, Ohio, assignors of 76.89% to R. E. MacKenzie and 23.11% to L. V. Bockius. Filed July 18, 1946. 2 claims. (Cl. 117-113.) Laminated corrugated board articles are impregnated in such a manner that the predetermined shape and dimensions of the articles are maintained.

2,552,643. Display package. Ersel C. Mulnix, Lake Bluff, Ill. Filed Dec. 7, 1945. 3 claims. (Cl. 229-16.) A display container incorporates a front cover flap to effect its closure.

2,552,871. Log barking apparatus having converging oscillatable water jets and means to proportionally vary center of convergence and amplitude of oscillation. Ernest C. Shaw, assignor to Allis-Chalmers Manufacturing Co., Milwaukee, Wis. Filed Nov. 18, 1948. 8 claims. (Cl. 144-208.) A hydraulic barker.

2,553,154. Method of waterproofing and flameproofing paper. Edward H. Voigtman and John C. Bletzinger, assignors to Paper Patents Co., Neenah, Wis. Filed Oct. 3, 1945. 5 claims. (Cl. 117-92.) Paper is treated with an aqueous emulsion of asphalt and then with an emulsion of a fire-inhibiting salt and a water-resistant wax.

2,553,227. Composite sheathing board and method of making same. Wilton W. Wesner, Rancocas, N. J. Filed Oct. 25, 1945. 10 claims. (Cl. 154-116.) A sheet of Celotex is disposed in the same plane between narrow strips of wood.

2,553,412. Molding fiber composition. Clark C. Heritage assignor to Wood Conversion Co., St. Paul, Minn. Filed Dec. 13, 1943. 4 claims. (Cl. 92-3.) Aspen chips are defibrated in the presence of a phenol-formaldehyde resin.

2,553,418. Article isolating shipping container. Gaylord Loth, assignor to The Hinde & Dauch Paper Co., Sandusky, Ohio. Filed Jan. 18, 1950. 3 claims. (Cl. 229-14.) The carton contains buffer members to support and protect a fragile article.

May 22, 1951

2,553,592. Coating apparatus for wrapping and cartoning machines. Fred W. Kucklinsky, assignor to Esther M. Kucklinsky, Montclair, N. J. Filed March 19, 1947. 14 claims. (Cl. 91-49.) An internally supplied glue applicator roll is arranged for skip-gap or surface application to a carton or package.

2,553,733. Machine for setting up corner locking trays. Ralph E. Acker, assignor to Container Corp. of America, Chicago, Ill. Filed Feb. 20, 1948. 8 claims. (Cl. 93-51.) Corner-locking containers are set up from blanks by the movement of a plunger into a throat.

2,553,740. Machine for covering knocked down cartons, Samuel Bergstein, Cincinnati, Ohio. Filed Aug. 31, 1946. 16 claims. (Cl. 93-54.) A machine wraps the tubular body of a window carton in a cellophane sleeve-like member.

2,553,803. Carton. Julius A. Zinn, Jr., Chicago, Ill. Filed March 25, 1947. 7 claims. (Cl. 229-37.) A siftproof carton is designed to resist crushing during the sealing operation.

2,553,804. Carton having end closures of substantially uniform thickness. Julius A. Zinn, Jr., Chicago, Ill. Filed March 28, 1947. 8 claims. (Cl. 229-37.) A siftproof carton has end closures of substantially uniform thickness which is adapted to be sealed without being crushed.

2,553,816. Pressure-sensitive adhesive and adhesive tape. Clarence J. Ebel, assignor to Minnesota Mining & Manufacturing Co., St. Paul, Minn. Filed Feb. 16, 1948. 10 claims. (Cl. 206-59.) The base of the adhesive is a rubbery copolymer of 2-ethylbutyl acrylate and ethyl acrylate.

2,553,840. Bendable clip box lock. Michael C. Andre, assignor to Gaylord Container Corp., St. Louis, Mo. Filed April 12, 1950. 3 claims. (Cl. 229-47.)

2,553,870. Box blank and box. Rolf A. Samsing, assignor to Associated Folding Box Co., South Boston, Mass. Filed June 29, 1950. 3 claims. (Cl. 229-35.) A one-piece box blank.

2,553,923. Wrapping paper comprising single-face corrugated board and integral fly webs. Ralph E. Lambert, Fort Wayne, Ind. Filed Sept. 11, 1948. 4 claims. (Cl. 206-59.) A roll of wrapping paper comprises single-face corrugated board and integral fly webs.

2,554,004. Mechanism and method for making boxes with attached handles and the like. Samuel Bergstein, Cincinnati, Ohio. Filed May 18, 1944. 36 claims. (Cl. 93-39.)

2,554,017. Electroresponsive recording blank. Harold R. Dalton, assignor to Timefax Corp., New York, N. Y. Filed Nov. 14, 1946. 7 claims. (Cl. 117-33.5). The blank incorporates a surface coating which is adapted to be removed in localized areas in response to applied electric signals.

2,554,027. Slitting device. Henry L. Haswell, assignor to Ecusta Paper Corp., Pisgah Forest, N. C. Filed July 23, 1946. 3 claims. (Cl. 164-70.)

2,554,034. Papermaker's felt. John P. Koester and Harold S. Comer, assignors to The Orr Felt & Blanket Co., Piqua, Ohio. Filed Aug. 18, 1948. 2 claims. (Cl. 139-426.) The wrap system of the woven felt is a cotton yarn and the filling system is a mixture of cotton and wool yarns which are laid in a regular repeating pattern.

2,554,035. Manufacture of combined paperboard and silicate-clay adhesives therefor. Robert L. Kreyling, assignor to Philadelphia Quartz Co., Philadelphia, Pa. Filed Sept. 3, 1946. 6 claims. (Cl. 154-33.05.) The preparation of a clay-silicate adhesive is described.

2,554,089. Structural covering material. Michele Croce assignor to Certain-Teed Products Corp., Chicago, Ill. Filed June 28, 1946. 2 claims. (Cl. 117-140.) A roofing felt comprises a resin-impregnated sheet of fibrous material.

2,554,137. Package with improved tear-tape. Drury R. Burton, assignor to Wingfoot Corp., Akron, Ohio. Filed July 9, 1947. 1 claim. (Cl. 229-87.)

2,554,157. Package. James E. Snyder, assignor to Wingfoot Corp., Akron, Ohio. Filed July 26, 1948. 1 claim. (Cl. 229-55.) A double-wall bag, each wall being composed of different wrapping materials.

2,554,190. Display carton. Russell J. Hennessey, assignor to Waldorf Paper Products Co., St. Paul, Minn. Filed Nov. 29, 1946. 12 claims. (Cl. 206-65.) A display carton for canned goods.

2,554,439. Modification of cellulose by boron trifluoride solution. Louis H. Bock, assignor to Röhm & Haas Co., Philadelphia, Pa. Filed April 22, 1948. 7 claims. (Cl. 8-116.) Paper is parchmentized by treatment with a 58 to 67% aqueous boron trifluoride solution at −5 to 30°C.

2,554,454. Process for laminating paper. George S. Casebolt, assignor to American Cyanamide Co., New York, N. Y. Filed June 22, 1946. 2 claims. (Cl. 154-138.) The adhesive consists of a mixture of starch and melamine-formaldehyde resin.

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2,554,574. Box forming machine. Arnold A. Kaehler and Enoch B. Johnson, Red Wing, Minn. Filed Aug. 4, 1945. 13 claims. (Cl. 93-36.4) A carton closing and sealing machine.

2,554,613. Cellular carton. Martin Burger, assignor to Shellmar Products Corp., Chicago, Ill. Filed July 28, 1945. 14 claims. (Cl. 229-28.) A die-cut multicompartment egg carton.

2,554,662. Process of glossing paper. William W. Cowgill, assignor to United States Rubber Co., New York, N. Y. Filed Aug. 16, 1947. 2 claims. (Cl. 117-65.) The paper is coated with a styrene-acrylonitrile or styrene-butadiene copolymer, dried, and pressed against a heated surface.

2,554,673. Method of glossing paper. William W. Cowgill, assignor to United States Rubber Co., New York N. Y Filed Dec. 15, 1949. 7 claims. (Cl. 117-65.) Paper is coated with a styrene-butadiene copolymer, dried, and the coating set by heating at 100° for about 35 seconds.

2,554,677. Foldable box. Louis C. Michetti, assignor to Andre Paper Box Co., San Francisco, Calif. Filed Aug. 16, 1947. 1 claim. (Cl. 229-35.) A folding box of the snap-open type.

2,554,722. Method of forming cylindrical containers. Harry F. Waters, New York, N. Y. Filed June 10, 1949. 4 claims. (Cl. 154.83.) A method for forming cylindrical paper containers with a multi-ply wall (one fluted ply and two liners).

2,554,814. Filter materials and process for making such materials. John B. Catlin and John C. Wollwage, assignors to Paper Patents Co., Neenah, Wis. Filed May 21, 1945. 9

claims. (Cl. 154-138.) A filter element is formed from several plies of creped tissue impregnated with phenol-formaldehyde resin.

2,554,899. Grease-proof paper and process of making the same. William W. Cowgill, assignor to United States Rubber Co., New York, N. Y. Filed Dec. 20, 1949. 4 claims. (Cl. 117-76.) Board is coated with a copolymer of butadiene and styrene and then with a mixture of a copolymer of styrene-acrylonitrile and one of butadiene-acrylonitrile.

2,554,934. Method of manufacturing structural insulation. Osborn Ayers, assignor to Johns-Manville Corp., New York, N. Y. Filed Nov. 2, 1945. 3 claims. (Cl. 92-21.) An asbestos insulating board incorporated sodium silicate and calcium chloride as a binder.

2,554,988. Apparatus for laminating materials. Robert F. Hollis, assignor to Alton Box Board Co., Alton, Ill. Filed Dec. 23, 1946. 4 claims. (Cl. 154-1.) A continuous press comprises a pair of endless bands having adjacent reaches sufficiently spaced to admit the material to be laminated.

2,554,989. Pulpwood chain sawing machine. Alfred J. Johnson, Ranier, and Foster D. McCormack, International Falls, Minn. Filed April 24, 1948. 7 claims. (Cl. 143-32.)

2,555,031. Container. Mary C. Fox and Charles C. Howes, assignors to The Davidson Chemical Corp., Baltimore, Md. Filed July 10, 1946. 2 claims. (Cl. 206-2.) A multilayer kraft paper bag for fertilizers.

2,555,057. Process for the production of paper coating adhesives. Laurence W. Porter and Frank H. Jewett II, assignors to S. D. Warren Co., Boston, Mass. Filed Dec. 10, 1947. 10 claims. (Cl. 260-17.3.) A mixture of 0.63 part soda ash and 84 parts melamine-formaldehyde is added to 280 parts tapioca or cassava starch in 520 parts water, heated 10 minutes at 205°F., the cooled solution is diluted with 415 parts of water and 200 parts of coating-grade calcium carbonate, and then with 800 parts coating clay in 430 parts water.

2,555,266. Method of printing and coating paper. Donald B. Bradner, assignor to The Champion Paper and Fiber Co., Hamilton, Ohio. Filed July 10, 1945. 19 claims. (Cl. 101-426.) A process which provides in one operation a printed, coated, cast-surface sheet suitable for labels.

June 5, 1951

2,555,315. Method of producing containers. Joseph P. Carroll, assignor to American Can Co., New York, N. Y. Filed July 5, 1945. 2 claims. (Cl. 93-36.) A liquidproof container has a tubular fiber body and a substantially flat end member adhesively secured to folded end portions of the body.

2,555,321. Facsimile recording paper. Harold R. Dalton and Austin G. Cooley, assignors to The Western Union Telegraph Co., New York, N. Y. Filed Aug. 8, 1941. 8 claims. (Cl. 204-2.) The paper base has a thin conducting layer of gas black and a binder (e.g., Methocel) and an overlying white facsimile coating containing zinc oxide.

2,555,337. Chemical furnace and recovery unit. Alexander L. Hamm, assignor to Combustion Engineering-Superheater, Inc., New York, N. Y. Filed April 11, 1945. 2 claims. (Cl. 23-262.) A self-sustaining unit for the recovery of heat and chemicals from waste liquors.

2,555,380. Container. Kimberly Stuart, Menasha, Wis., and Allen B. Wilson, Evanston, Ill., Wilson assignor to Elizabeth R. B. Stuart, Menasha, Wis. Filed Jan. 21, 1946. 5 claims. (Cl. 229-3.5.) A spirally wound tubular container for foods and liquids has a substantially continuous metal foil lining.

2,555,401. Roofing and the manufacture thereof. George A. Fasold and Harold W. Greider, assignors to The Philip Carey Manufacturing Co., Lockland, Ohio. Filed Dec. 28, 1944. 20 claims. (Cl. 117-92.) A roofing felt containing glass fibers is impregnated with asphalt.

2,555,506. Bonded laminae and method of making same. Donald H. Powers, assignor to Monsanto Chemical Co., St. Louis, Mo. Filed July 8, 1947. 17 claims. (Cl. 154-138.) Paper sheets are impregnated with a colloidal solution of silica and the dried sheets are impregnated with a liquid resin containing styrene, ethylene glycol, and maleic anhydride.

2,555,526. Pour seal structure for cartons. Julius A. Zinn, Jr., Chicago, Ill. Filed July 15, 1948. 9 claims. (Cl. 229-17.) A liquid-tight container incorporates a pour seal structure which includes a bellows-type top closure.

2,555,536. Method of coating continuous webs of paper or the like. Harry W. Faeber, assignor to Time, Inc., New York, N. Y. Filed Nov. 17, 1948. 8 claims. (Cl. 117-111.) A method is proposed for applying several layers of coating material to a paper web.

2,555,581. Carton closure. Robert M. Dunning, assignor to Waldorf Paper Products Co., St. Paul, Minn. Filed July 18, 1947. 6 claims. (Cl. 229-43.) A glued-end carton is provided with triangular supporting struts which resist outward sealing pressure against the carton end.

2,555,655. Folding box. Claude D. Painter, San Francisco, Calif. Filed Nov. 19, 1945. 4 claims. (Cl. 229-32.) A method of erection or opening folding boxes.

2,555,700. Container with sealing lid. John G. O'Neil, assignor to O'Neil-Weinhagen, St. Paul, Minn. Filed April 8, 1946. 1 claim. (Cl. 229-5.5.) A cylindrical container for foods has a tight-fitting lid.

2,555,718. Collapsible suitcase. Joseph H. Vineberg, Ottawa, Canada. Filed Jan. 2, 1946. 7 claims. (Cl. 190-43.)

2,555,719. Collapsible box. Joseph H. Vineberg, Ottawa, Canada. Filed April 17, 1946. 7 claims. (Cl. 206-7.) A one-piece garment-carrying box.

2,555,758. Wrapping machine. Frank W. Noble and Donald C. Baker, assignors to The Robinson Waxed Paper Co., Limited, Bristol, England. Filed May 4, 1948. 7 claims. (Cl. 93-3.) A means for wrapping disk-shaped articles (e.g., biscuits).

2,555,794. Well-drilling fluid. Rudolf A. Henkes, assignor to Shell Development Co., San Francisco, Calif. Filed Oct. 18, 1948. 4 claims. (Cl. 252-8.5.) Tall oil is an emulsifying agent.

2,555,844. Carry bag. Edward M. De Rouville, assignor to United States Envelope Co., Springfield, Mass. Filed June 26, 1947. 4 claims. (Cl. 229-54.) The extensions of the walls of a paper carry bag form a hand grip.

2,555,983. Clasp-type closure for envelopes. George H. Matthiesen, Chicago, Ill. Filed June 4, 1947. 4 claims. (Cl. 229-78.)

2,556,011. Method of making a cellular structural panel and product. Donald L. Swayze and Augustus H. Nicholls, assignors to Chrysler Corp., Highland Park, Mich. Filed May 5, 1947. 3 claims. (Cl. 154-33.05.) A structural panel comprises a facing skin (plywood, cardboard, etc.) and an intermediate cellular structure of phenolic resin-impregnated paper.

2,556,032. Roller coating machine. Harry W. Faeber, assignor to Time, Inc., New York, N. Y. Filed Oct. 6, 1947. 19 claims. (Cl. 91-50.) A roll-coating machine is provided with an improved quick-release mechanism for interrupting the coating process when the paper web breaks.

2,556,069. Bottle carton. Grover C. Currie and Walter A. Becker, assignors to Dacam Corp., Charlotte, N. C. Filed Nov. 28, 1947. 1 claim. (Cl. 224-45.) The end walls of the carton can be collapsed between the side walls.

2,556,078. Transfer and method of coating therewith. Carleton S. Francis, Jr., assignor to American Viscose Corp., Wilmington, Del. Filed May 18, 1945. 5 claims. (Cl. 154-95.) The paper base of a flexible transfer sheet is coated with phenol-formaldehyde resin and has a temporary coating of a vinyl resin.

2,556,088. Collapsible cellular carton. William H. Inman, assignor to Bloomer Bros. Co., Newark, N. Y. Filed May 8, 1948. 1 claim. (Cl. 229-28.) The carton is adaptable to packaging fragile articles (eggs, cake, fruit, and the like).

June 12, 1951

2,556,262. Method of coating paper. Harry W. Faeber, assignor to Time, Inc., New York, N. Y. Filed Nov. 16, 1946. 3 claims. (Cl. 117-111.) The coating composition on the transfer rolls is maintained at the dew point of the surrounding atmosphere by the internal cooling of one roll or by the additon of moisture to the atmosphere.

2,556,321. Liner and receptacle. Harvey R. Denton, assignor to Moist-R-Proof Container Co., San Francisco, Calif. June 24, 1946. 1 claim. (Cl. 229-14.) A waterproof liner (regenerated cellulose or chlorinated rubber) for a food carton is provided with a collar of semirigid material.

2,556,333. Process for the manufacture of a novel binding agent for core sand from sulphite lye. Franz R. Moser, assignor to Oel- und Chemie-Werk A.G., Hausen, Switzerland. Filed Feb. 11, 1948. 6 claims. (Cl. 22-188.) Spent sulphite liquor is heated with an excess of sulphuric acid until a marked increase in viscosity is produced.

2,556,334. Process for the manufacture of a novel binding agent for core sand from sulphite lye. Franz R. Moser, assignor to Oel- und Chemie-Werk A.G., Hausen, Switzerland. Filed Feb. 11, 1948. 8 claims. (Cl. 22-188.) A chemical (e.g., ammonium sulphate) is added to the spent sulphite liquor and the mixture is heated until a marked increase in viscosity results.

2,556,341. Collapsible paperboard bottle carrier. William A. Ringler, assignor to The Gardner Board and Carton Co., Middletown, Ohio. Filed Oct. 16, 1947. 4 claims. (Cl. 229-52.) A carrier is formed of single-faced board, printed on only one side.

2,556,568. Container, Harry S. Aikman, Port Huron, Mich. Filed Nov. 9, 1946. 3 claims. (Cl. 229-6.) A shipping container can be converted into a baking pan.

2,556,606. Flexible oil acid modified alkyd resin wrinkle coating composition and method of making. William A. Waldie, assignor to New Wrinkel, Inc., Wilmington, Del. Filed March 12, 1946. 4 claims. (Cl. 260-22.) The composition contains glycerin, phthalic anhydride, castor oil acids, a drying oil, and a drier.

2,556,638. Paper container for pastries. Frederick O. Wickersham, Chicago, Ill. Filed Nov. 3, 1947. 6 claims (Cl. 206-45.17.)

2,556,661. Convertible shipping carton and self-feeder. Warren C. Rendall and Fred L. Rushing, assignors to Gaylord Container Corp., St. Louis, Mo. Filed Jan. 10, 1949. 5 claims. (Cl. 229-7.) A shipping carton for poultry feed can be converted to a trough-type self-feeder.

2,556,707. Convertible shipping carton and self-feeder. Warren C. Rendall and Fred L. Rushing, assignors to Gaylord Container Corp., St. Louis, Mo. Filed Dec. 31, 1948. 4 claims. (Cl. 229-7.) A shipping container for poultry feed incorporates a folded self-feeding insert.

2,556,778. Collapsible carrier with means for maintaining expanded condition. William A. Ringler, assignor to The Gardner Board and Carton Co., Middletown, Ohio. Filed Feb. 15, 1949. 8 claims. (Cl. 229-28.) A carrier has means for maintaining its erected condition.

2,556,898. Urea-formaldehyde-sulphoxylate resin for high wet-strength paper. Alfred Brookes and Frederick L. Hudson, assignors to American Cyanamid Co., New York, N. Y. Filed Dec. 16, 1946. 12 claims. (Cl. 92-21.) Paper is sized with 0.25 to 25% by weight of a water-soluble sulphoxylated condensation product of urea and formaldehyde.

2,556,915. Reinforced carton. Reynolds Guyer, assignor to Waldorf Paper Products Co., St. Paul, Minn. Filed June 19, 1946. 1 claim. (Cl. 229-37.) A glued-end carton is constructed with two reinforced opposed walls.

2,557,011. Corrugating roll lubricator. Albert F. Shields, assignor to S&S Corrugated Paper Machinery Co., Inc., Brooklyn, N. Y. Filed March 17, 1948. 3 claims. (Cl. 154-30.)

2,557,017. Reducing paper tubes in diameter, William F. Stahl, Kenilworth, Ill. Filed Oct. 1, 1945. 3 claims. (Cl. 93-94.) An apparatus for reducing the diameter of a portion of a laminated paper tube.

2,557,025. Denture liner of bibulous paper impregnated with vinyl resin. Maurice B. Brand, Chicago, Ill. Filed Sept. 9, 1949. 6 claims. (Cl. 260-31.8.) A denture liner consists of thin bibulous paper (e.g., yoshino paper) impregnated with a vinyl chloride-vinyl acetate resin and a vinyl acetate resin.

June 19, 1951

2,557,071. Process of making a plywood product. Robert M. Boehm, assignor to Masonite Corp., Laurel, Miss. Filed Oct. 12, 1945. 5 claims. (Cl. 154-133.) Wood veneer is hydrolyzed under the Masonite conditions, arranged in stacks, and subjected to heat and pressure.

2,557,112. Combined container and cover. Earl L. Kaffer, Joliet, Ill. Filed Feb. 6, 1947. 2 claims. (Cl. 229-43.) A closure for a package of deodorant powder, the outer layer of which is cellophane over an inner layer of cotton netting or wire cloth.

2,557,174. Apparatus for pulping paper stock. Edwin Cowles, assignor to The Cowles Co., Princeton, N. J. Filed Jan. 26, 1949. 10 claims. (Cl. 92-26.) In a pulper a small impeller creates and maintains adequate circulation of the stock in the tank.

2,557,290. Dispensing container. Wallace M. Houldsworth, Royal Oak, Mich. Filed July 27, 1946. 20 claims. (Cl. 229-7.) A dispensing container for matches, toothpicks, and the like.

2,557,299. Urea-formaldehyde paper-treating composition. James C. Lehr, Denis O'Flynn, and Reade Y. Tompson, assignors to E. I. du Pont de Nemours & Co., Wilmington, Del. Filed March 9, 1950. 14 claims. (Cl. 260-29.4.) A high wet-strength paper is produced by adding 0.5 to 2% of a diglycolic acid-modified urea-formaldehyde condensation product.

2,557,455. Flotation of ilmenite ores. Samuel P. Moyer, assignor to American Cyanamid Co., New York, N. Y. Filed March 12, 1948. 1 claim. (Cl. 209-166.) Tall oil is used.

2,557,504. Cutout clearer for perforated containers. Raymond D. Holmes, assignor to Fleming Specialty Co., Charlotte, N. C. Filed April 17, 1950. 4 claims. (Cl. 93-36.)

2,557,914. Folding carton and opening flap therefor. Otto M. Miller, assignor to Owens-Illinois Glass Co., Toledo, Ohio, Filed Jan. 8, 1948. 12 claims. (Cl. 229-51.) A folding carton has a pull-up or opening flap.

June 26, 1951

2,558,054. Purification of cellulosic materials. Mervin E. Martin and Daniel L. Sherk, assignors to Celanese Corp. of America, New York, N. Y. Filed March 16, 1949. 2 claims. (Cl. 8-105.) A sulphite pulp is subjected to an initial alkaline cook, chlorinated, and given another alkali cook at a temperature above 160°; the second cook reduces the pentosan content.

2,558,169. Valved bag. Charles V. Brady and August F. Ottinger, assignors to Bemis Bro. Bag Co., St. Louis, Mo. Filed Sept. 24. 1948. 9 claims. (Cl. 150-9.) A textile or paper-lined textile bag has an insert-type valve sleeve.

2,558,287. Paper cup and handle therefor. Walter E. Amberg, assignor to Lily-Tulip Cup Corp., New York, N. Y. Filed Nov. 19, 1945. 5 claims. (Cl. 229-52.) A method of attaching a handle to a double-wrapped paper cup.

2,558,392. Moisture indicator for webs. John S. Seney, assignor to E. I. du Pont de Nemours & Co., Wilmington, Del. Filed May 14, 1949. 3 claims. (Cl. 161-1.) The indicator is adapted for the continuous measurement of the electrical resistance of a portion of the web which, in turn, is a function of its moisture content.

2,558,456. Box making, filling, and closing apparatus. Frank D. Palmer, assignor to Package Machinery Co. East Longmeadow, Mass. Filed April 22, 1948. 17 claims. (Cl. 93-55.1.) Method for packaging dry powdered, granular, liquid, or semi-liquid materials.

2,558,543. Tall oil separation process. James W. Davis and Harold M. Spurlin, assignors to Hercules Powder Co., Wilmington, Del. Filed Oct. 18, 1949. 14 claims. (Cl. 260-97.6.) A method of fractionation of resin and fatty acids with gasoline and isopropyl alcohol.

2,558,618. Bottle carrier. Samuel N. Lebold, assignor to Morris Paper Mills, Chicago, Ill. Filed Jan. 8, 1948. 5 claims. (Cl. 229-52.) The carrier has integral reinforcing elements.

2,558,634. Paper impregnating or saturating composition. Jay J. Uber, assignor to E. I. du Pont de Nemours & Co., Wilmington, Del. Filed Feb. 9, 1949. 5 claims. (Cl. 117-155.) A wet paper web is impregnated with a mixture of rubber latex and the sodium salt of lignin.

2,558,689. Variable diameter winder shaft. Clarence W. Miller, assignor to D. J. Murray Manufacturing Co., Wausau, Wis. Filed March 5, 1948. 2 claims. (Cl. 242-72.)

2,558,712. Bottle carrier. Marshall I. Williamson, assignor to National Folding Box Co., Inc., New Haven, Conn. Filed Dec. 24, 1949. 4 claims. (Cl. 224-48.)

2,558,713. Bottle carrier. Marshall I. Williamson. assignor to National Folding Box Co., Inc., New Haven, Conn. Filed Dec. 24, 1949. 4 claims. (Cl. 224-48.)

2,558,714. Bottle carrier. Marshall I. Williamson, assignor to National Folding Box Co., Inc., New Haven, Conn. Filed Dec. 24, 1949. 9 claims. (Cl. 224-48.) The handle of a carrier is constructed of a single ply of kraft paperboard.

2,558,715. Multicompartment folding carton. Marshall I. Williamson, assignor to National Folding Box Co., Inc., New Haven, Conn. Filed April 21, 1950. 7 claims. (Cl. 224-48.) A multicompartment carrier embodies individual cells formed by webs extending between the side walls.

Paper and Packaging Specifications

U. S. Government Specifications

Following is information from B. W. Scribner, National Bureau of Standards, Chairman, Technical Committee on paper and Paper Products, Federal Specifications Board, on Federal specifications of recent issue. This is a revision.

Paper, Teletype, Roll and Tape, UU-P-547d

Extensive changes have been made in the specification. It is now confined to papers on which the message is typed with ink. The oiled perforator type formerly included has been deleted and will be issued as a separate specification because of considerable expansion of the requirements. The other important changes are in the requirements for the roll type. A 100% bleached chemical wood-fiber paper has been added, the chemical wood-fiber content of the other grade has been reduced to 40%, many changes have been made in the requirements for the physical properties, and rolls with an increased number of plies have been added.

(A revision of the Federal Specifications Index, revised to Jan. 1, 1951, has been issued under the new title, "Federal Specifications and General Services Administration Specifications Index." This and Federal specifications may be obtained on application accompanied by money order, coupon, or cash to the Superintendent of Documents, Government Printing Office, Washington 25, D. C. The price of the Index is 35 cents, and the price of the specifications is 5 cents, each.)

UTILIZATION OF SULPHITE WASTE LIQUOR

A Bibliography of the Literature from July, 1943, to December, 1950

Compiled by H. BORDEN MARSHALL and A. MARGARET JOHNSON

In the production of sulphite pulp approximately 50% of the wood is discarded as lignin and other organic materials in the form of sulphite waste liquor, representing a tremendous loss to Canadian pulp mills. The problem of finding uses for these by-products has been a matter of growing concern to the industry, not only to obtain the maximum commercial value per pound of wood used, but, perhaps even more urgently, to avoid the unfavorable conditions, such as stream pollution, that often result from the disposal of this material.

Over the years a great deal of research has been done on various aspects of this general problem, and much valuable information has been obtained. As the work has progressed it has become even more necessary that the results be summarized and catalogued in an orderly fashion so as to avoid repetition and duplication of effort, to give an indication of those aspects of the problem that appear most promising, and to provide a background of information for new research projects that will be undertaken in the future.

The early literature on the subject up to 1918 was ably reviewed by D. Johnsen and T. W. Hovey in Bulletin No. 66 of the Forestry Branch, Dominion Department of the Interior, Ottawa. This was followed in 1943 by a survey of the literature for the period 1917–43 by B. E. Mead, R. F. Patterson, and G. F. Pepper under the auspices of the Ontario Paper Co. Ltd. Copies of this summary were distributed through the Canadian Pulp and Paper Association, Montreal.

Increasing activity in this field of research in recent years has resulted in a voluminous literature, and the need for a further review has become apparent. On the recommendation of the Wood Chemistry Subcommittee,* Advisory Committee on Forestry, this project has been sponsored by the Research Council of Ontario, the work being done by H. B. Marshall and Miss A. M. Johnson of the Ontario Research Foundation. This covers the literature for the period from July, 1943, to December, 1950, and provides the third volume of a summary of the literature on the subject from its beginning to the present time.

The primary intention of this bibliography is to provide the research worker in lignin chemistry with a convenient file of abstracts on the subject of sulphite waste liquor. The information is based to a large extent on material published in *Chemical Abstracts*. An attempt has been made to include all the articles on sulphite waste liquor which appeared and to make the abstracts as complete as space would permit. The progress made during this period has not been summarized nor have the results of the individual investigations been evaluated. These are tasks for experts in the respective fields.

The abstracts are arranged chronologically under the same general uses as in the preceding volume by Mead, Patterson, and Pepper.

Report No. 4-4-50, Research Council of Ontario, 39 Queens Park, Toronto, Ont., June, 1950.

* Members of this Subcommittee are: H. B. Marshall (Chairman), Ontario Research Foundation; G. A. Ledingham, National Research Council Regional Laboratory, Saskatoon; G. H. Tomlinson II, Howard Smith Paper Mills Ltd., Cornwall; R. R. McLaughlin, Department of Chemical Engineering, University of Toronto; G. A. Adams, National Research Council, Ottawa; and F. Bender, Forest Products Laboratory, Ottawa.

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1. INITIAL TREATMENT OF SULPHITE WASTE LIQUOR

A. Evaporation Processes

ROSENBLAD, C., *Papier-Fabr.* 40: 165–171 (1942); cf. *C. A.* 36: 2411 (1942); *C. A.* 37: 5233 (1943).
A discussion of modern mill problems concerning the evaporation of sulphite waste liquor with flue gases or steam.

v. LASSBERG, J., *Papier-Fabr.* 40: 193–200, 208–208 (1942); cf. *C. A.* 36: 6014 (1942); *C. A.* 37: 5233 (1943).
An illustrated address on the concentration and burning of sulphite waste liquor including a brief historical résumé and 24 references.

TÄNZLER, K. H., *Gesundh. Ing.* 65: 433–434 (1942); *Chem. Zentr.* 1943. I: 2160–2161; *C. A.* 38: 1110 (1944).
The Kunze method of concentrating sulphite waste liquor by agitation (warm air in the colder months) is discussed.

HIÄRNE, E. H., *Finnish Paper Timber J.* 21: 250, 252–253 (1939); *Chem. Zentr.* 1943, II: 586; *C. A.* 38: 4438 (1944).
A survey of the recent literature leads to the conclusion that the best method for recovery of sulphite waste liquor is the completely automatic Bergson system.

RAMÉN, T., U. S. pat. 2,343,027 (Feb. 29, 1944); *C. A.* 38: 4804 (1944).
A pretreatment is employed which involves injecting partially concentrated liquor into hot gases in a finely divided state such that they are dehydrated by the gases to a dry powder. This powder is separated from the gases and the latter passed in direct heat exchange with cooled water so that the steam contained in the gases condensed. The heat of evaporation is thus liberated and absorbed by the cooling water. The water, after heat exchange with the gases, is passed in indirect heat exchange with a portion of the sulphite waste liquor which is continuously circulated in a circuit. The liquor at one zone of the circuit is continuously vacuum concentrated and a dilute waste liquor added to the circuit at a point ahead of the zone of concentration. Liquor is withdrawn from the circuit at a point on the other side of the zone of concentration for injection into the hot gases. An arrangement of apparatus is described.

Bergson, C. R., *Finnish Paper Timber J.* 25: 349–356 (1943); *Chem. Zentr.* 1943, II: 2027; *C. A.* 38: 5669 (1944).
Data are given for the amount and density of the cooking liquor, the density of the wash water, and that of the total liquor. Preliminary concentration can be carried out with steam or by freezing. The freezing process has the advantage of the lack of incrustation in the evaporators and also improved properties of the concentrated liquor for further utilization. The dried lignin can be used as a fuel. The concentration can be combined with alcohol manufacture in that the liquor is first concentrated to a suitable concentration for fermentation, the slops are further concentrated and the residue is dried to powder form.

Kunze, W., *Deut. Wasserwirt.* 1942: 123–126; *Wasser u. Abwasser* 40: 92–93 (1942); *C. A.* 39: 1536 (1945).
For the concentration of sulphite waste liquor by evaporation, air at velocities of 1 to 4 meters per second is blown over the sulphite waste liquor as it flows slowly through zinc pans. Liquor from the digesters is added to the first pans and recycled until a concentration corresponding to 28.5° Baumé is attained.

v. Lassberg, J., *Papier-Fabr. Wochbl. Papierfabr.* 1944: 123–127; cf. *C. A.* 37: 5233 (1943); *C. A.* 39: 1758 (1945).
An illustrated report on the concentration and burning of sulphite waste liquors dealing with developments during 1942–43.

Schmidt, E., *Deut. Wasserwirt.*, 1942: 378–380; *Wasser u. Abwasser* 40: 180 (1942); *C. A.* 39: 2198 (1945).
The calculations of Kunze (*C. A.* 39: 1536 (1945)) were too optimistic, and his process is no more economical than those usually employed.

Müller, C., and Scholz, P., Ger. pat. 744,869 (Nov. 25, 1943); *C. A.* 39: 2878 (1945).
When sulphite liquor from beechwood and similar woods is evaporated, the scale-forming material, notably calcium sulphate, is removed before the liquor gets to the evaporator. This is accomplished by superheating the liquor in a directly or indirectly heated pressure kettle. The heating is carried out at the original pH of the liquor or at an acidity reduced to pH 4.5.

Firing, W., Norw. pat. 69,764. (Nov. 18, 1945); *C. A.* 40: 2044 (1946).
The description of an apparatus for evaporating liquids, particularly waste liquors, from the manufacture of cellulose.

Setterwall, F., *Pulp Paper Mag. Can.* 46: 1004–1005 (1945); *C. A.* 40: 2304 (1946).
A brief outline of recent developments in sulphite waste liquor evaporation in Sweden. An evaporator has been designed which is not built of tubes but of steel plates which form two equal channels, one for the liquor and the other for the steam. After operating for a certain time, the flow is reversed so that the liquor channel becomes the steam channel and vice versa. This removes the calcium sulphate scale which deposits on the heating surface, solution of the scale being hastened by the fact that the condensate is acid.

Zak, H., *Papier-Fabr. Wochbl. Papierfabr.* 1944: 194–198; *C. A.* 40: 3897 (1946).
A lecture dealing with the recovery of waste liquor with special reference to modern evaporator systems. Diffusers and multiple-stage washing operations are illustrated schematically. The relationship between specific gravity of waste liquors from beech and spruce and their total solids, sugar content, and potassium permanganate numbers is shown graphically.

Wurz, O., *Papier-Fabr. Wochbl. Papierfabr.* 1944: 230–232; *C. A.* 40: 3897 (1946).
An illustrated, descriptive article outlining the work of other engineers and discussing an experimental technique for withdrawing liquors as completely as possible within a given time.

Edling, G., *Pulp and Paper Ind.* 19: 58, 60 (1945); *C. A.* 40: 4213 (1946).
The Ramén (cf. *C. A.* 30: 7732; 31: 2411) and the Rosenblad (*C. A.* 36: 2411) systems of evaporation of sulphite waste liquor are discussed.

Nyman, C., *Svensk Papperstidn.* 49: 73–83 (1946); *C. A.* 40: 5564 (1946).
The heat economy obtainable by evaporation of calcium-base sulphite waste liquor through indirect heating to a concentration at which it can be burned is intimately connected with the problems arising from incrustation of the heating surfaces. Nyman presents a detailed review of the various factors governing the incrustation and the various theoretical and practical means of reducing it. Calcium sulphate is the dominating incrusting component. The physical conditions under which the various modifications of this salt appear are given in graphs and tables with many references to previous studies. Hägglund (*C. A.* 28: 1528) has established that formation of sulphate ion is part of the sulphite pulp cooking process. Thus, the presence of considerable amounts of calcium sulphate in the sulphite liquor is unavoidable. Organic components in the liquor are protective colloids and bring about supersaturation of calcium sulphate. This favorable effect is increased with higher pH toward neutrality. Mathematical expressions are given for incrustation build-up, but the equations obtained are of doubtful value because of the many uncertainties in the factors involved. Examples are given of experimentally determined increase in incrustation with acid sulphite liquor, alcohol-fermented sulphite liquor, and for evaporation under different temperatures. Various means of pretreating the waste liquor to reduce incrustation are discussed. Addition of surface-active agents (i.e., sodium hexametaphosphate, citric acid, tanning extract from oak) in the small amounts which the economy of the process permits does not have a favorable effect. A recently patented process reduces the calcium sulphate considerably through a not too expensive coprecipitation with calcium sulphite under very specific conditions. A brief description of the Grewin-Lindberg procedure (*C. A.* 40: 2304) is given, whereby the evaporation takes place in a heat exchanger in which the channels for sulphite liquor and those for steam can be reversed by a simple mechanical manipulation requiring but a few minutes. Thus, after certain intervals, the steam is passed through the channels previously occupied and incrusted by sulphite liquor and the acid steam condensate dissolves and cleans the incrusted surfaces. This affords a practical means of removing incrustants before they will cause too great loss in heat transfer and without interrupting the operation of the equipment.

Lur'e, M. Y., *Isvestiya VTI (Vsesoyuz. Teplotekh. Inst. im. Feliksa Dzerzhinskogo)* 15, No. 3: 4–7 (1946); *C. A.* 40: 5565 (1946).
The sulphite waste liquor at the Archangel pulp mill is sent to an alcohol plant where the liquor is fermented to ethanol. The residue contains approximately 5% of solids. It is then concentrated in two stages, in the first of which the liquid is sprayed into boiler waste gases having a temperature of 170°. In the second stage, the liquid is sprayed into flue gases having a temperature of 700 to 900°. The liquid is concentrated to 50% solids and in this state is used as a bonding agent for foundry sand. The waste gases are scrubbed free of ash and dust and are discharged into the atmosphere. Details of this installation are given.

Waenerlund, L., *Svensk Papperstidn.* 49: 200–202 (1946); *C. A.* 40: 6813 (1946).
Relative to incrustation difficulties occurring on the evaporation of sulphite waste liquor, the factors which affect the sulphate content of the liquor were studied. The raw cooking liquor contained sufficient amounts of sulphate to give incrustation. Investigation showed that formation of sulphate during the preparation of cooking acid can be greatly reduced by adding small amounts of sulphite waste liquors to the make-up water, since such liquor evidently inhibits the oxidation of sulphur dioxide. Considerable quantities of sulphur dioxide partly from loosely combined sulphur dioxide, are driven off if sulphite waste liquor is evaporated somewhat prior to neutralization, and in the latter step aeration should be eliminated.

Byström, S., Swed. pat. 104,783 (June 16, 1942); Swed. pat. 105,702 (Oct. 13, 1942); *C. A.* 40: 7627 (1946).
An apparatus for evaporating sulphite waste liquor.

Persson, A. R., and Naucler, J. O., Swed. pat. 102,092 (July 22, 1941); *C. A.* 40: 7627 (1946).
A process for the multiple-stage evaporation of sulphite waste liquor.

Persson, A. R., and Naucler, J. O., Swed. pat. 105,401 (Sept. 1, 1942); *C. A.* 40: 7627 (1946).
A process for the evaporation of acid and alkaline waste liquors obtained in the manufacture of cellulose.

Sillen, L. G., *Svensk Papperstidn.* 49: 387–392 (1946); *C. A.* 41: 2892 (1947).
The incrusting substances frequently deposited on the heating surfaces of evaporators for calcium-base sulphite waste liquor seriously reduce heat transfer of such equipment, which then requires frequent shutdowns for costly mechanical or chemical cleaning. Chemical and x-ray investigations of numerous samples from large-scale evaporators have shown such incrustants to consist most often of calcium sulphate. Calcium sulphite and silicate very seldom deposit on heating surfaces and the former is subsequently easily removed. The three modifications in which calcium sulphate can exist (anhydrous, hemihydrate, and dihydrate) are discussed as to their solubility and conversion into one another by temperature. The dihydrate deposits below 100°, whereas the hemihydrate forms above 100° and incrustants constituting the latter are more difficult to remove. Efforts to reduce incrustation on evaporating calcium-base sulphite waste liquor center on reducing the sulphate content. Previously known analytical methods for sulphate content as well as that of Döring (*C. A.* 36: 267), have given erroneous results. Sillen outlines a new analytical method which he

claims gives consistent and sufficiently accurate results for comparison between sulphate content of the liquor and incrustation in evaporators. In this method the sulphite liquor is added to formaldehyde and acetic acid, whereby sulphur dioxide compounds are combined as an aldehyde addition complex and thus prevented from oxidation to sulphate. The original sulphate ions are then precipitated as barium sulphate in the cold solution and centrifuged. Several tables give the sulphate content of samples from different mills of their cooking acid, sulphite waste liquor before and after neutralization, and before and after distillation of alcohol obtained on fermentation. Various means of decreasing the sulphate content in these different stages are discussed.

KARLBERG, R., *Svensk Papperstidn.* 49: 412–417 (1946); *C. A.* 41: 2893 (1947).

The Grewin-Lindberg system (cf. Brauns, *C. A.* 37: 6890; 38: 6554) for concentrating and burning sulphite waste liquor, which is operated in mill scale at Holmens Bruk pulp mill, is described. The system (cf. Setterwall, *C. A.* 40: 2304: Nyman, *C. A.* 40: 5564) involves the patented principle of periodically exchanging the steam and liquor channels in the heat exchangers to redissolve the incrustants deposited on the heating surfaces without interruption of the operation. The plant is described and illustrated in pictures and drawings. It operates with four effects in the temperature range from 120 to 55° and is designed to evaporate 8.6 tons per hour of 15% sulphite liquor to 2.6 tons per hour of 50% sulphite liquor with 1.6 tons per hour steam. The sulphite liquor processed receives no pretreatment prior to the evaporation. The strongest liquor draining from the digesters has 15% solids, but more liquor is furnished to the evaporation plant by including some sulphite liquor from displacement washing in the digester whereby the over-all liquor concentration for evaporation is 13 to 14%. The periods between exchange of steam and liquor channels in the heat exchangers were brought up to 8 to 12 hours and the heating surfaces were kept clean this way in the second and fourth effects. Burned liquor solids deposited in the inlet and outlet of the first effect on which is received the most concentrated liquor. By reducing the superheat in the steam for this effect, caking of the burnt solids was reduced but not eliminated and must be dissolved off periodically with concentrated nitric acid. In the third effect gypsum could not be eliminated without periodic mechanical cleaning. Gypsum deposits also occurred in pipe lines and circulating pumps between the effects and had to be cleaned periodically or interchanged with spare parts, but this could be done quickly and in normal shutdown periods. Gypsum deposited in the bottom of the expansion vessels, mainly in the third effect, from which twice a year over 1000 pounds gypsum (dihydrate) was removed. The plant operated with one man per shift; repair and cleaning required 4 to 5 man-days per week. Some operating data for 1943–45 are given, but these are not conclusive because the sulphite mill operation was irregular during this period. The liquor, concentrated to 50% solids, was sprayed under 6 atm. and at 80° into a boiler, with 300 sq. meters heat surface on a bed of wood fuel and with a feed of 1.5 to 2 tons liquor per hour. Sooting of tube banks and superheaters increased when sulphite liquor was first used; thus, soot cleaning was required for coal firing every 2 to 3 months, with hog fuel and bark every 1 to 2 months and the latter plus concentrated sulphite liquor every 1 to 2 weeks; such cleaning required 6 to 8 man-hours. The boiler, prior to burning the concentrated sulphite liquor, had been equipped with a "BAHCO" dynamic flue-gas filter and no complaints were heard about fly ash resulting from the sulphite liquor combustion. Some special tests indicated that, after starting up with wood fuel, a boiler can be maintained by 100% loading with 50% sulphite liquor, and the data indicated good combustion efficiency.

ULFSPARRE, S., *Svensk Papperstidn.* 49: 383–386 (1946); *C. A.* 41: 2893 (1947).

During the last few years the adverse fuel situation in Sweden, both as regards cost and availability, has created interest in the problem of economical evaporation and burning of sulphite waste liquor. Ulfsparre gives an account of the results thus far achieved by a committee which, on recommendation of the sulphite committee of the Central Laboratory of the Cellulose Industry, was appointed to investigate the technical and economical aspects. Means of minimizing heat loss caused by the incrusting liquor components were investigated, not only by improving evaporator design, but also by conducting the cooking operation and liquor-collection procedures that more suitable sulphite liquor will result. With standard operations liquor from dissolving pulp gives less incrusting difficulties than from strong pulp. Efficient stripping of sulphur dioxide from the liquor will reduce incrustation. Pre-evaporation of the liquor prior to alcohol fermentation will accomplish this and was further studied as was the simultaneous evaporation and alcohol distillation of fermented sulphite liquor. Various procedures worked out in different mills for collecting strong liquor and pulp washings without undue dilution and heat loss are being obtained for a later report. Reuse of dilute sulphite liquor in cooking acid is being investigated. The problem of handling and disposing of the large amount of ash resulting from burning sulphite liquor is considered. Corrosion from sulphur trioxide in flue gases is also given attention in large-scale operating plants. Recovery of sulphur in combination with burning of sulphite liquor is believed unlikely when calcium-base cooking is used.

RAMEN, T., *Svensk Papperstidn.* 49: 418–422 (1946); *C. A.* 41: 2894 (1947).

A description of the Ramén process for concentrating, drying, and burning sulphite waste liquor as installed at Hornefors, Sweden, in 1944. The sulphite liquor which previously has been fermented for alcohol enters the process with 9.4% solids at 105° in amounts of 38.4 tons per hour. It passes through four-effect evaporators with temperatures of 100, 85, 65 and 35°. Heat to the last effect is furnished not only from the third effect but also from the last scrubbing of the flue gas. The liquor passes from the fourth effect with 40% solids to the spray drier. Hot flue gases are drawn from the boiler and ejected with the concentrated liquor into a drying chamber with cyclone separator for the dried powder which is then ground and blown into the boiler much like powdered coal. From the cyclone the flue gas and steam formed from drying the concentrated liquor passes through a filter and into a concentrator charged with liquor from the fourth effect. Not only is further heat from the flue gas utilized to concentrate liquor prior to spray drying, but fine dust not held back by the filter is retained in the concentrator. The flue gas and steam leave the latter at 80° and pass through a water scrubber then to a stack at 50°. The outflowing scrubber water can furnish heat by exchange to the fourth-effect evaporator. Practical experience with the system in large-scale operation has shown only minor difficulties from incrustation and those occur mostly in the fourth effect where waste liquor concentration is the highest. Clean and efficient heating surfaces in the evaporators are maintained by constructing them from cold-rolled, acid-resistant steel with a very smooth surface, and by using rapid liquor circulation. For each effect, the heater is constructed in sections, each of which can be cleaned without interrupting the total operation. Nevertheless, incrustation is always a threatening factor for the efficiency and any possible remedy should be used to furnish liquor with reduced incrustation tendencies. Recent experiments with preliminary heating of incoming sulphite liquor to a higher temperature seem promising. Incrustants separate in this step and carry through the concentrating steps in suspension without depositing on heating surfaces. Combustion of the dried powder under the boilers gives no difficulties and is complete, provided the powder is finely ground. This fuel has 10% ash which tends to collect on the tubes, but is easily removed by ordinary soot blowers. Preliminary observations have shown no danger of corrosion of the economizer from sulphur dioxide when dry sulphite liquor powder is fired. Complete data on the net economy accomplished by the described process are not yet available for publication but Ramén asserts that: "the problem of evaporation of sulphite waste liquor can now be assumed solved from the heat-technical operating as well as the economical side."

HEDBORG, F. O., Swed. pat. 117,579 (Nov. 5, 1946); *C. A.* 41: 4647 (1947).

Before evaporation and burning of sulphite waste liquor, a solution of sodium sulphide is added together with a stream of carbon dioxide and the calcium carbonate formed is removed. In a modification, the sodium sulphide solution is added in two stages and after neutralizing to pH 5, the sugar in the liquor is fermented to alcohol. In the second stage, the remainder of the sodium sulphide is added at pH 9 and the solution evaporated and burnt. The sodium sulphide in the smelt is used again.

BRUNES, B., SAMUELSON, O., AND ULFSPARRE, S., *Svensk Papperstidn.* 50, No. IIB: 29–33 (1947); *C. A.* 41: 7113 (1947).

Five systems of evaporation of sulphite waste liquor are compared:

(1) Evaporation of wash *in vacuo* with five effects.
(2) Combination of pressure and vacuum evaporation of the wash in six effects, withdrawing steam for the alcohol plant after three effects.
(3) Combination of pressure and vacuum evaporation of the original liquor, neutralized liquor, and wash in six effects, withdrawing steam for the alcohol plant after three effects.
(4) Combined evaporation and alcohol distillation of fermented liquor in a six-effect vacuum evaporation.
(5) Pressure evaporation in three effects and withdrawal of steam after the second effect to machine room; pressure steam to digesters and alcohol plant.

These systems have not been used under practical conditions

of operation. The heat balance is given for each system and also the heat consumption in the concentration of sulphite waste liquor under certain assumed conditions. The problem of incrustation by calcium sulphate is discussed especially as regards the effect of temperature on the solubility of the various solid phases of calcium sulphate and its hydrates.

Samuelson, O., *Svensk Papperstidn.* 50: 239–244 (1947); *C. A.* 42: 1055 (1948).
Pressure evaporation of sulphite waste liquor affords better heat economy than evaporation under vacuum, but presents a more difficult problem owing to incrustation on the heating surfaces. Samuelson describes a study of the nature of and conditions under which incrustations are formed. A two-stage pilot plant pressure evaporator was used in which the liquor was first preheated to 163° for varying lengths of time, then evaporated in a Kesstner-type combination heat exchanger and expansion vessel which could be operated at 112 and 145°. The Grewin-Lindberg channel exchange principle (*C. A.* 40: 2304, 5564) was used in the heat exchanger. Analysis showed that loosely combined sulphur dioxide was driven off in the pressure heater, as well as in the evaporator and, to a considerable extent, was found in the condensate recombined with volatile carbonyl compounds as α-oxysulphonic acids. During pressure heating and evaporation, loosely combined sulphur dioxide will neither form insoluble sulphite nor oxidize to sulphate. Pressure heating of neutralized (i.e., alcohol-fermented) liquor causes rapid precipitation of calcium sulphite, some of which redissolves when the temperature decreases. Owing to the lower solubility of calcium sulphate at increasing temperatures, as much as 80% of this compound may precipitate out on the preheating at 163°, but this effect is also a function of heating time. Decreasing the temperature in the subsequent evaporation at lower temperatures will redissolve some calcium sulphate. These findings are discussed in relation to formation of sulphate and solubility of calcium sulphate during sulphite pulping, pulp washing, and the most suitable arrangment for temperature and heating steps in the liquor evaporation. Sometimes silica has been found as an incrustation component. A practical method for the analysis of silica in sulphite waste liquor was worked out. For the liquor investigated, the silica content was only in the range of 0.5 to 1.5 mg. per liter. A schematic drawing is given of the experimental equipment and the analytical data are given in six graphs.

Sillén, L. G., *Svensk Papperstidn.* 50: 339–345 (1947); cf. *C. A.* 41: 2892 (1947); *C. A.* 42: 1055 (1948).
With the recently developed rapid and reliable method for determination of the sulphate content of sulphite waste liquor (*C. A.* 41: 3294), the incrustation problem has been further studied. Analyses of the sulphate content of cooking acid, sulphite waste liquor removed before and after neutralization, and before and after alcohol fermentation and distillation are given in a table for samples from eight different sulphite mills. Unavoidably sulphate forms during the cooking process in such amounts that the sulphate content of the fresh cooking liquor is of little importance. With calcium-base cooking liquor, calcium sulphate precipitates on the pulp during the cook as the temperature increases, and thus sulphate in solution actually decreases. The precipitated calcium sulphate will in part redissolve in the pulp-washing process. Thus, dilute sulphite waste liquor from such washing has a high sulphate content owing to this and also owing to some oxidation of sulphur dioxide to sulphur trioxide. The solubility of calcium sulphate anhydrite and calcium sulphate hemihydrate in sulphite waste liquor has been investigated at elevated temperatures. Slow conversion from the comparatively soluble hemihydrate takes place at about 127° above which the solid phase is anhydrite which is much less soluble. This points out the need for frequent cleaning of heat-exchange surfaces while incrustants consist mainly of hemihydrate. It is practically unavoidable that the concentrated sulphite waste liquor is supersaturated with respect to anhydrite, but the sulphate separating out in check liquor is as a sludge which does not appear to adhere to heating surfaces. Reference is made to Samuelson's (preceding abstract) experiments on decreasing incrustations in the earlier stages of evaporation through a preliminary high temperature heating stage. Analytical data are given.

Bergström, H. O. V., and Trobeck, K. G., Swed. pat. 118,913 (May 27, 1947); *C. A.* 42: 1738 (1948).
The formation of hard deposits in the digestion of cellulose by the sulphite process and in the evaporation of the sulphite waste liquor is prevented by adding 1 to 10 grams of sodium hexametaphosphate per cubic meter to the cooking acid or to the waste liquor prior to evaporation.

Ekeberg, G. E.. Swed. pat. 119,130 (June 25, 1947); *C. A.* 42: 1738 (1948).
The layout of a plant is described whereby sulphite waste liquors are evaporated and steam is simultaneously recovered for power generation.

Rosenblad, C., *Northeastern Wood Utilization Council Bull.* 19: 123–135 (1948); *C. A.* 42: 4745 (1948).
A discussion of the Rosenblad system for evaporation of sulphite waste liquor.

Markham, A. E., and McCarthy, J. L., *Paper Trade J.* 126: 70–72 (1948); *C. A.* 42: 5664 (1948).
No nitrogen is removed during the vacuum evaporation of ammonium sulphite waste liquor containing up to 4.9% ammonia determined by Kjeldahl method, up to 4.35% "free ammonia," and 1.56 to 1.81% loosely bound sulphur dioxide (as sulphur). Some of the nitrogen in the liquor is combined in such a manner that it is not readily released by boiling under mildly alkaline conditions. The liquor contained up to 0.42% calcium, at least a part of which resulted from the calcium in the wood (western hemlock); some of the calcium precipitated as calcium sulphate during the evaporation. Steam stripping of the ammonium sulphite waste liquor proceeds more slowly than in the case of the calcium liquors and leads to a marked reduction in loosely combined sulphur dioxide, a decrease in acidity, and substantially no change in the reducing sugar concentration. Cation-exchange treatment (Ionex No. 2) replaces nearly all the ammonium and calcium ions with hydrogen.

Brunes, B., *Svensk Papperstidn.* 51: 336–342 (1948); cf. *C. A.* 41: 7113; *C. A.* 43: 399 (1949).
The amounts and concentration of sulphite waste liquor used for alcohol fermentation, as well as for evaporation and burning, are the most important factors for these utilization processes. By means of practical average data from many Swedish plants and certain assumptions, the relationship between dry solids and tons liquor per ton pulp for strong pulp and rayon pulp cooks are graphically presented. From these curves and values for prices of coal and alcohol and plant cost for vacuum and pressure evaporation, curves are given for the relationship between economic gain and tons of liquor used for each ton of pulp. These graphs (which are also summarized in a table) demonstrate the important relationship between cost of coal and the amount of liquor (per ton pulp) which it is economical to process. With low coal price, more liquor can be pulled for alcohol production than for evaporation and burning but, with the present high price for coal in Sweden, the difference in the liquor volumes which are economical to process per pulp ton with the several alternate processes is relatively small.

Thuillier, H. M., *Papeterie* 70: 323–324 (1948); *C. A.* 43: 399 (1949).
A descriptive article discussing the possibility of applying destructive distillation (not combustion) to the wastes from the pulp industry.

Beath, L. R., and Hill, H. S., U. S. pat. 2,453,775 (Nov. 16, 1948); *C. A.* 43: 1188 (1949).
Dilute sulphite waste liquor is concentrated to at least 50% solids for use as a fuel and the recovered sulphur dioxide is suitable for use in the pulp-cooking process. The sulphite liquor near its maximum concentration is passed between electrodes in a heating chamber maintained at a pressure in excess of the maximum vapor pressure of the liquor. The electric current passing through the liquor is so regulated that not more than 200 B.t.u. per pound of liquor is generated. The heated liquor is passed to a flash chamber where a pressure less than the vapor pressure of the liquor is maintained, which causes the water and sulphur dioxide to be vaporized. A part of the concentrated liquor is fed back into the heating chamber together with additional dilute sulphite waste liquor.

Nelson, B. A., and Mannbro, N. V., Swed. pat. 121,440 (Apr. 13, 1948); *C. A.* 43: 3617 (1949).
Sulphite waste liquors, particularly calcium bisulphite waste liquors, are used to extract the soda melt obtained in the working up of the black liquor from an alkaline cellulose process, and the solution so obtained is causticized and reused in the known manner for the manufacture of pulp.

Smith, H. L., *Pulp Paper Mag. Can.* 50, No. 9: 84–86 (1949); *C. A.* 43: 8677 (1949).
Design calculations are presented for a sulphite waste liquor evaporator operated on the thermal compression principle. A pilot-scale unit had a power consumption of 160 kw.-hr. per 1000 gallons of distillate. A commercial unit driven by electricity should consume 111 kw.-hr. per 1000 gallons of distillate or 107 per ton of 50% solids. Operated by steam, the unit would use 1580 pounds steam per 1000 gallons, or 1520 pounds per ton.

Beath, L. R., and Hill, H. S., *Pulp Paper Mag. Can.* 50, No. 8: 89–94 (1949); *C. A.* 43: 8677 (1949).
A description is given of the development of an electrical evaporator for sulphite waste liquor. In the final pilot-scale design, the liquor flows through a perforated grounded electrode, past a high-voltage electrode and a second grounded electrode, and through a valve to a steam separation chamber. Power input can be regulated by controlling steam bubble generation within

the electrode chamber. The liquor in the evaporator is kept at constant concentration by appropriate addition of weak and withdrawal of strong liquor. In the pilot evaporator, voltages were varied from 700 to 1100, power input up to 65 kw. Waste liquor decomposition was 10% when operating at 150 p.s.i. This increases the heat value per pound of concentrated liquor, but decreases total heat recovery and hampers operation somewhat through viscosity increase.

Brauns, O., *Svensk Papperstidn.* 50, No. 11B: 61–62 (1947); *C. A.* 43: 8677 (1949).
A brief description of the proposed six-effect pressure evaporation system, in which superheated steam at 240° is put into a third effect, which is connected with a vessel for separating gypsum. A schematic drawing and a table show steam input and water removal, starting from 10.9% and finishing with 50% solids in the liquor processed.

Folger, H. H., Herbolzheimer, F., Stinchfield, R. M., and Jenness, L. C., *Tappi* 32: 389–393 (1949); *C. A.* 43: 9441 (1949).
A series of experiments were made in a tower spray drier and in a Bowen laboratory drier to determine the over-all heat-transfer coefficient for sulphite waste liquor. The coefficient K was calculated from $Q = KV\Delta tm$, where Q = B.t.u. per hour; K = (B.t.u. per hour)(cu. ft.)(°F.); V = volume of chamber in cubic feet; Δtm = log mean temperature difference between wet and dry bulb temperature of drying gas. The optimum values of K were found to decrease with increasing solids content of the feed. Values of K ranged from 4.6 at 45% solids to 10 at 8% solids.

Aspergen, J. A., Swed. pat. 124,414 (Mar. 22, 1949); *C. A.* 43: 9448 (1949).
An apparatus for the high-temperature evaporation of sulphite waste liquor.

Butler, W. T., *Pulp Paper Mag. Can.* 50, No. 11: 108–121 (1949); *C. A.* 44: 1698 (1950).
A review of the evaporation and burning of calcium-base sulphite waste liquor with 51 references.

Grewin, F. W., and Lindberg, S. G., U. S. pat. 2,490,750 (Dec. 6, 1949); *C. A.* 44: 1706 (1950).
To remove scale formed on the walls of apparatus where sulphite waste liquor is being concentrated by heat exchange with steam, the paths of flow of the solution and the steam are altered at suitable intervals, such as approximately 30 hours, so that the channel which forms the passage for the solution during one period will form the passage for the steam during a following period, and vice versa in alternate succession. Preferably, with the steam is mixed some SO_2 liberated from the solution; this gives the steam a somewhat acid character which considerably increases its ability to dissolve the scale. The heat-exchange apparatus best adapted to this operation is of the spiral or the plate type with channels for steam and solution of the same shape and dimensions.

Tyden, A. E., U. S. pat. 2,490,759 (Dec. 6, 1949); *C. A.* 44: 2154 (1950).
Deposition of scale in heat exchangers may be controlled by providing passages for fluid flow on both sides of the wall surfaces, using a chemically active scale solvent in the heating fluid, and intermittently reversing the passages used for the different fluids. For example in evaporating sulphite waste liquors a solution of H_2SO_3, or SO_2-bearing steam or vapors to which a solution of sodium hexametaphosphate is added, may be used for the scale-removing agent. After a period, the paths of flow are reversed in order to clean the opposite side of the walls.

Samuelson, O., *Svensk Papperstidn.* 52: 283–289 (1949).
By use of the recently described reliable method for the determination of sulphate in waste liquor (*C. A.* 42: 1055), the precipitation of $CaSO_4$ from sulphite waste liquor was studied as a function of time, temperature, and pH. Results reported in graphs and tables show that the maximum precipitation is at pH about 3.5 and the precipitation increases at higher temperatures. Suspended crystals of $CaSO_4$ increase precipitation velocity by acting as crystallization nuclei. The total solids content of the sulphite waste liquor in the range of 8 to 20% has only a slight influence on the amounts of $CaSO_4$ precipitated. Similar studies in two different types of large-scale evaporating plants for sulphite waste liquor show less precipitation of $CaSO_4$ from neutralized fermented liquor than from the original acid liquor, which is in accord with lab findings. By x-ray investigations it was established that anhydride forms at evaporating temperatures higher than 130° and hemihydrate resulted when the evaporating temperature was 100 to 130°.

Nyman, C. H., U. S. pat. 2,484,046 (Oct. 11, 1949); *C. A.* 44: 3257 (1950).
The pH of sulphite waste liquor is adjusted to 6 to 6.5 by adding Na_2CO_3. This forms a suspension of CaS in the solution. During the heat-exchange process, CaS and $CaSO_3$ are co-deposited as a scale which may be removed by using weak acids.

Franklin, J. N., and Goddard, H. O., *Pulp Paper Mag. Can.* 51, No. 3: 139–46 (1950); *C. A.* 44: 6120 (1950).
A pilot plant was constructed and successfully operated for the concentration of sulphite waste liquor by using steam at 200 to 250 p.s.i. The steam produced from the waste acid at 100 p.s.i. can be used to reconvert the boiler condensate to low-pressure steam for drier operation, etc. By using two heaters (three recommended for commercial practice), continuous operation could be maintained. Heater scale was removable with acid condensate. Solids in the concentrated waste liquor of 60% were obtained. Detailed economic considerations are given.

Elgee H., Craig, D., and Russell, J. K., *Pulp Paper Mag. Can.* 51, No. 3: 178–189; *C. A.* 44: 6120 (1950).
The thermal compression cycle has been successfully applied in a pilot plant to the evaporation of sulphite waste liquor. It is necessary to clean the evaporator for 4 hours with acid condensate after 20 hours' operation. No improvement in scaling was obtained by heat-pretreatment of the liquor. Foam inhibitors added to the feed made the scale slightly more easily removable. Energy requirements amounted to 22 kw.-hr. per 1000 pounds evaporated. This would be reduced in a full-scale evaporator and would amount to about 106 kw.-hr. per ton of 50% solids concentrate produced from 10% solids feed.

Brunes, B., *Svensk Papperstidn.* 52: 529–532 (1949); *C. A.* 44: 7536 (1950).
The use of sulphite waste liquor as a fuel, either as a liquor containing about 50% solids or a powder, presents problems because of its high ash (7 to 15%) and sulphur content. The dried powder burns even in well-cooled boilers without auxiliary fuel but gives an ash which tends to deposit on the heating surfaces, particularly superheaters. The liquor containing 50 to 55% solids requires additional fuel (oil or powdered coal) or noncooled furnace walls to maintain the necessary temperature for complete combustion. Ash from this fuel is nonadhering and the greater part is collectable in mechanical separators. Both fuels produce some SO_3 in the flue gases, thereby raising the dewpoint from 2 to 40°. Loss of combustible material in the ash is still a problem which may possibly be solved by construction of improved injecting nozzles.

Rosenblad, C., *Pulp Paper Mag. Can.* 51, No. 6: 85–94 (1950); *C. A.* 44: 8646 (1950).
A detailed description of the Rosenblad system for evaporation of sulphite waste liquor.

Scholander, A., *Svensk Papperstidn.* 52: 35–38 (1950); *C. A.* 44: 8646 (1950).
The amounts and concentration of ash and SO_2 in flue gases from burning sulphite waste liquor are compared with those from coal and oil. Typical composition of ash from sulphite waste liquor: $Fe_2O_3 + Al_2O_3$ 7, $CaSO_4$ 36, $CaCO_3$ 42, CaO 14, and CaS 1%. Incorrect values for C in flue ash are obtained by direct ignition; a correct analytical method is given. In an effort to eliminate the air-polluting effect of SO_2 and fly ash, experiments were made on wet scrubbing of the flue gas with the ash; 100 or 50% of the SO_2 can theoretically be retained in the scrubber dependent on whether or not oxidation is avoided. Similar tests on a large scale gave only 30% SO_2 absorption, with 60% oxidation. Consideration of such scrubbing is discussed from the standpoint of elimination of air pollution vs. the problem of disposing of the scrubber liquor.

Central Laboratory of the Swedish Cellulose Industry, Analysis Committee, *Svensk Papperstidn.* 53: 13 (1950); *C. A.* 44: 8652 (1950).
In a glazed porcelain or Pt dish, about 50 to 60 mm. outside diameter, a sample is weighed out which will give an evaporation residue of 0.3 to 0.4 gram. The sample is placed in a constant temperature drying oven at 105° for 24 hours, cooled in a desiccator for 1 hour, and weighed to the nearest 0.1 mg. The residue is expressed in percentage by weight of the sample and reported to one decimal.

Scholander, A., *Svensk Papperstidn.* 53: 95–102 (1950); *Norsk Skogind.* 4: 63–71 (1950); *C. A.* 44: 9148 (1950).
By use of the terms and calculation of Nilsson (*C. A.* 36: 3044), the relationship between liquor solids, volume, and percentage of total quantity is discussed. Economic concentration depends on pulp quality, alcohol and fuel prices, and evaporation cost. A table gives the economic concentrations under various conditions. A model with continuous flow is used in calculating the liquor-recovery process and the results are compared with reported mill-operating data. The principal factors affecting favorable liquor recovery by drainage and replacement from digester are temperature, uniform packing of chips and a high and narrow digester. A well-defibered pulp, such as that obtained by blowing the cooked chips into pit or diffuser, will improve liquor recovery. Graphs illustrate correlation between factors important to liquor recovery.

Coldwell, F. H., and Kennedy, E. H., *Combustion* 22, No. 1: 51–55 (1950); *C. A.* 44: 9133 (1950).
Concentrated sulphite waste liquor (52% solids) and coal were burned successfully in a 125,000 pounds steam per hour boiler; mechanical atomizing and steam atomizing nozzles are used. Test results and details on fly ash control and effect of liquor on equipment are given.

Bender, F., *Pulp Paper Mag. Can.* 51, No. 8: 73–76 (1950); *C. A.* 44: 11090 (1950).
Passage of a direct current through sulphite waste liquor causes the deposition of Ca compounds at the cathode. In small-scale experiments on a continuous rotary cell it was possible to reduce the Ca content from 0.92 to 0.586 gram per 100 ml.

Kennedy, E. H., and Coldwell, F. H., *Paper Mill News* 73, No. 31: 12, 14–17 (1950); *C. A.* 44: 11090 (1950).
Sulphite liquor, concentrated to 52% solids, was burned successfully under a boiler with an efficiency of 74%, as compared with 83% for the same unit when fired with coal.

B. Precipitation Processes

Rieche, A., and Hilgetag, G., Ger. pat. 734,471 (Mar. 18, 1943); *C. A.* 38: 1366 (1944).
The two-stage precipitation process is described in which a protein yeast is grown on the sulphite liquor and then ligninsulphonic acid precipitated in the well-known manner.

Norsk Hydro-Elektrisk, Kvaelstofaktieselskab, Norw. pat. 64,263 (Dec. 15, 1941); *C. A.* 40: 737 (1946).
Organic substances are precipitated from sulphite waste liquor by adding a small quantity of selenium, e.g., 1 to 10 mg. of selenium dioxide per liter, and heating under pressure, e.g., at 150° and 6 to 7 atm. for 10 minutes. The organic matter separates as a solid mass.

Bergström, H. O. V., Trobeck, K. G., and Tydén, H., Swed. pat. 115,813 (Feb. 12, 1946); *C. A.* 40: 6816 (1946).
Sulphite waste liquor, from which sulphites have been wholly or partially recovered, is fractionally precipitated by means of oxides or hydroxides of alkali metals, alkaline earth metals, iron or aluminum, separately or together, at a temperature not above 75°. Precipitation takes place in stages and is not carried beyond the stage in which almost pure salts of ligninsulphonic acid are obtained. The remaining solution may be treated with further amounts of oxides or hydroxides to precipitate the remaining salts of ligninsulphonic acids together with a certain amount of carbohydrate material. A still further quantity of oxide or hydroxide may then be added to precipitate the carbohydrates. The precipitate containing both ligninsulphonic acid salts and carbohydrates may be treated with a dilute solution of the oxide or hydroxide whereby the carbohydrate is dissolved.

Baum, M., Bard. J. W., Salvesen, J. R., and Brabender, G. J., *Paper Trade J.* 126, No. 8: 130, 132–135 (1948); *C. A.* 42: 2433 (1948).
Experimental sulphite pulping of eastern hemlock was carried out to establish the influencing factors on yield of lime-precipitated lignin from the resulting sulphite waste liquors. The pulping variables investigated included unreacted and combined sulphur dioxide, cooking time, and maximum temperature. Results for pulps having permanganate numbers of 16.9, 12.7, and 8.4 show that the highest yield of precipitated lignin is obtained when processing liquors from pulps having the lowest permanganate number. When liquor from pulps of low permanganate number is processed, the recovery of lignin is practically unaffected by the composition of the cooking liquor or maximum temperature over the range studied. When pulp of higher bleachability is produced, lignin recovery is more favorable at lower concentrations of unreacted sulphur dioxide and, to a lesser extent, at lower combined sulphur dioxide in the cooking acid. Tables are given showing the percentage distribution of lignin in pulp, irrecoverable waste liquor, lime-precipitated fraction, and effluent from recovery process. These results are shown for typical sulphite cooking conditions producing pulps with three different bleachabilities. Effects of the same cooking variables on yield and bleachability of eastern hemlock pulp are given in accompanying curves which show trends in agreement with results reported by others.

Diotti, G., Ital. pat. 422,330 (June 13, 1947); *C. A.* 43: 4854 (1949).
Increasing quantities of one of several mineral salts, such as ammonium sulphate, are added to sulphite waste liquor, which results finally in the precipitation of the ligninsulphonates in the solid state, while the solubility of the pentoses present is hardly affected, so that the salt solutions are more and more enriched in pentoses. Successive heatings of these solutions above 100° convert the pentoses into 2-furfuraldehyde under technically and economically favorable conditions. The apparatus is illustrated.

Slavik, J., and Borisek, R., *Chem. Zvesti* 1: 149–159 (1947); *C. A.* 43: 7227 (1949).
Organic substances in sulphite waste liquor are coagulated by heating with sulphuric acid (14.6 grams per liter) for 1 hour at 150°. Hydrochloric, nitric, and oxalic acids are less effective; phosphoric, boric, silicic, formic, acetic, and tartaric acids are without effect. Coagulation is accelerated by aluminum, chromous, and stannous ions, and is retarded by sulphuric ions. Concentration of the liquor facilitates coagulation.

Slavik, J., and Borisek, R., *Chem. Zvesti* 1: 299–305 (1947); cf. *C. A.* 43: 7227; *C. A.* 43: 8677 (1949).
In the precipitation of organic matter from sulphite waste liquor by pressure, the methoxyl group splits off the aromatic ring of the lignin. The free ortho and para positions are activated toward the liberated phenolic group and lignin is suitable for further aldehyde condensation and the formation of a three-dimensional macromolecule. The chemical constitution of lignin is explained as the formation and decomposition of macromolecules from phenol-aldehyde condensation products.

Nyman, C. H., Swed. pat. 128,210 (May 9, 1950); *C. A.* 9150 (1950).
Sulphite waste liquor is treated hot with a precipitant for sulphites, e.g., lime, in slight excess over that required to neutralize the liquor, and before, during, or after this addition, free SO_2 is passed in so that a substantial proportion of the $CaSO_4$ present in the liquor is precipitated simultaneously with the sulphite, and the residual liquor is slightly acid, e.g., pH 6 to 6.5.

C. Miscellaneous Processes

(a) Concentration by Freezing

Ax, G. B., Grunewald, K. H., Wiberg, O. A., and Öman, E., U. S. pat. 2,315,762 (Apr. 6, 1943); *C. A.* 37: 5288 (1943).
An apparatus is described for freezing out and separation of crystals from solutions such as concentrated sulphite waste liquors. The process involves freezing out part of the solvent to form crystals which are floated upwardly in a vessel containing the solution to a point of discharge from the vessel, washing and removing the crystals at this point of discharge, then withdrawing the solution freed of crystals from the vessel.

Öman, E., Swed. pat. 98,962 (May 28, 1940); *C. A.* 40: 778 (1946).
A process is described in which substances are purified by freezing an aqueous solution of the substance and subjecting the still fluid mixture of solution and ice crystals to intermittent pressure and washing. The wash liquor is continually removed and fresh frozen mixture added. The process is applicable to the separation of salt from sea water and the concentration of sulphite waste liquor.

(b) Fractionation by Adsorption on Ion Exchange Resins

Samuelson, O., *Svensk Papperstidn.* 46: 583–586 (1943); cf *C. A.* 37, 2573; *C. A.* 38: 2487 (1944).
The adsorption of the acids of sulphite waste liquor has been studied by the use of an organic cation exchanger (organolite) with an artificial resin base. The capillaries of the anion exchanger are so narrow that they permit only acids of low molecular weight to penetrate into the exchanger and be absorbed by it. When a number of pure acids of low molecular weight were compared, it was found that the amount of acid held by the exchanger increased as the molecular weight of the acids decreased. If the acids liberated from sulphite waste liquor are passed through a filter of such an organolite which is saturated with hydroxyl ions, the greater part of the ligninsulphonic acid will pass through it. The acids absorbed have a low methoxyl and sulphur content and consist largely of carboxylic acids. Elementary analyses and neutralization curves indicate that the acids absorbed consist of aldonic acids, sugar sulphonic acids, and ligninsulphonic acids. The acids can be separated from each other to a certain extent.

Tyler, R. G., U. S. pat. 2,392,435 (Jan. 8, 1946); *C. A.* 40: 2254 (1946).
Sulphite waste liquors which do not develop scaling troubles upon evaporation to 45 to 65% solids are obtained by passing the warm calcium or magnesium-base liquor through an acid-resistant base-exchanger such as Zeo-Karb. Thus, 350 ml. of a 10% liquor of pH 3.58 and 4.72% calcium content (dry basis) was passed through a stainless steel tube containing 76 grams of dry-base exchanger which had been previously conditioned with sodium chloride solution. A rate of 10 ml. per minute and a temperature of 180°F. gave 100% removal of calcium. The treated liquor was then evaporated to 45 to 65% solids without difficulty and was burned to recover sodium carbonate and sodium sulphate or sodium carbonate and sodium sulphide depending on the conditions of the furnace. Sea water may be used for partially regenerating the base exchanger.

After 170 cycles with liquors having pH values of 1.37 and 3.58, the exchanger suffered no physical deterioration, no loss in weight, and no loss of ability to remove calcium.

(*c*) *Fractionation by Dialysis*

Ögland, N. J., *Svensk Papperstidn.* 47: 288–291 (1944); *C. A.* 38: 5669 (1944).
A prerequisite for the manufacture of valuable products from sulphite waste liquor, such as those from hydrogenation, plastics, etc., is a lignin as free as possible from carbohydrates. The components of sulphite waste liquors can be separated by dialysis. A continuous process of separation was tried using an Alophan (cellophane-type) membrane. The following factors were varied: the entrance velocity of the sulphite liquor and the water, the concentration of sulphite liquor and its temperature. The highest efficiency (calculated per unit area of membrane) was obtained when the concentration and temperature of the sulphite liquor were high and the velocity of the water was three times that of the sulphite liquor. The chief drawback is the short life of the membrane. Otherwise cellophane has proved to be a suitable material for separating the lignin and carbohydrate components comparatively rapidly.

Hiester, N. K., McCarthy, J. L., and Benson, H. K., *Paper Trade J.* 126, No. 16: 58–61 (1948); *C. A.* 42: 3953 (1948).
Direct batch dialysis of sulphite waste liquor is a readily carried out procedure which can be used to obtain purified calcium ligninsulphonates in quantities sufficient to permit their evaluation for various utilization applications on a laboratory scale. Other salts or ligninsulphonic acids can be obtained by cation-exchange procedures by using appropriate resins. Analyses are given of products dialyzed for 213 to 335 hours, and preliminary data are given for the density of the ligninsulphonic acid and its calcium and sodium salts, as well as their surface tension and specific viscosity.

Peniston, Q. P., and McCarthy, J. L., *J. Am. Chem. Soc.* 70: 1324–1328 (1948); cf. *C. A.* 42: 3953; *C. A.* 42: 4745 (1948).
Dialysis of known aqueous mixtures of glucose and sucrose under four different flow conditions shows that, although the free diffusion rate for glucose is 1.37 times that for sucrose, the ratio of the dialysis rate coefficients is 1.64. This indicates a filtering action by the membrane for molecules as small as sucrose. Apparently the dialysis rates for these nonelectrolytes are not dependent on flow rate. Only a small change, if any, occurred in the porosity of the membrane during the entire period. Experiments with glucose and the sodium salt of *p*-methyl benzene sulphonic acid show that, although the two substances are of about the same molecular weight, the electrolyte dialyzed considerably faster than the nonelectrolyte and the dialysis rate coefficient of the electrolyte increases with flow rate. Two samples of sulphite waste liquor (analyses given) were studied with five different times for dialysis. The dialyzed liquors and dialyzates were analyzed for total solids, methoxyl, total reducing substances, and sulphur. The ligninsulphonic acids can be isolated in a high degree of purity in about 65 to 80% yield by exhaustive continuous dialysis of the waste liquor. The dialyzable ligninsulphonates are believed to have molecular weights of less than 200 and appear to vary in quantity depending on the conditions used during the pulping process. Two nondialyzable ligninsulphonates from different commercial sources are found to have nearly the same empirical composition when calculated to a sulphur and ash-free basis. This composition is in agreement with the concept of lignin as a polymer of guaiacyl-oxygenated propane structural units.

(*d*) *Purification by Removal of Inorganic Materials*

Vloeberghs, A., Belg. pat. 444,722 (Apr. 30, 1942); *C. A.* 39: 620 (1945).
A reducing agent capable of partially absorbing the coloring matter in the presence of sulphur dioxide is introduced into sulphite waste liquor, and the mixture is agitated while maintaining the temperature below 50°.

Danielsson, G. J. D., U. S. pat. 2,380,504 (July 31, 1945); *C. A.* 39: 5490 (1945).
Sulphite waste liquor is treated continuously with a neutralizing agent and the neutralized liquor is subjected to centrifugal action to remove the sludge; the clarified liquor is suitable for fermentation. The sludge is washed and the recovered sulphite liquor and neutralizing reagent are returned to the reagent tanks.

Braun, K., Ger. pat. 741,416 (Sept. 23, 1943); *C. A.* 40: 466 (1946).
The heavy metals are precipitated with potassium ferrocyanide and calcium and magnesium with sodium or potassium carbonate. The precipitation is carried out conveniently in a colloid mill or similar apparatus. The precipitates are removed and the liquor is bleached with hyposulphurous acid in the presence of a small quantity of hydrogen peroxide. This process for purification and bleaching applies equally to crude, concentrated, and fermented sulphite liquor.

2. BINDING MATERIAL

Rainus, R. N., *Liteinoe Delo* 12, No. 3: 10–12 (1941); *Chem. Zentr.* 1942, II: 451; *C. A.* 37: 6613 (1943).
Of materials used in mold and core mixtures, linseed oil has the greatest tendency to gas evolution, sulphite waste liquor the least. The greatest volume of gas is evolved in the first minute of heating. Two mixtures, one containing sand 75, bentonite 5, peat 20, dextrin 2, sulphite liquor 3, and one containing sand 75, bentonite 5, iron filings 20, dextrin 2, sulphite liquor 3, evolve large amounts of gas, which must be considered by providing for good mold ventilation. A mixture containing sand 77, reworked mold sand 20, fire clay 13, evolves about 5 liters gas per kilogram of mixture. The evolution stops after the first minute and the mixture can be considered practically as gas-free.

Grushevskii, N. V., *Liteinoe Delo* 12, No. 6: 32 (1941); *Chem. Zentr.* 1943 I: 80–81; *C. A.* 38: 2912 (1944).
A mixture formulated for cores of complicated design consisted of 100 parts quartz sand, 4 parts sulphite waste liquor (sp. gr. 1.3), and 1.5 parts linseed oil or 4 parts molasses (sp. gr. 1.2). A mixture of sufficient firmness and plasticity was obtained at a moisture content of 0.4 to 0.7%. Mixing was done in a mixer, samples were taken every 10 minutes during mixing, 10 samples in all. The moisture content was found to vary progressively with the period of mixing from 3.5 to 0.4%, the resistance to pressure from 0.03 to 0.29 kg. per sq. cm., and the permeability from 185 to 250 cm. per minute. To shorten the time of preparation the mixture can be made from dry or even warm sand and sirup or sulphite liquor of higher specific gravity. At present, such a mixture is prepared in 15 to 25 minutes with the moisture content at the start being 1 to 1.5%. The mixture possesses very good molding properties and can be used for the production of cores of complicated form. The drying of the cores is done at 200 to 210° for 6 to 10 hours, depending on the dimensions.

Axt, F., Belg. pat. 444,172 (Feb. 28, 1942); *C. A.* 39: 487 (1945).
Cellulose fibers are added to sulphite waste liquor as a core binder, to increase the plasticity of the sand and especially the resistance to collapse of the green cores. They burn when the casting is poured so that the cores lose all consistency and are easily removed.

Roll, F., and Arland, A., *Giesserei* 31: 110–113 (1944); cf. *C. A.* 38: 2287 (1944); *C. A.* 39: 4823 (1945).
A description of the procedures used for testing drying binders made from sulphite waste liquors, starch, potato meal, dextrin, coal-tar pitch, bitumens from brown coal and petroleum, clay, and sand.

Piette, G., and Demers, G., *Roads and Bridges* 83, No. 9: 70–73, 114–118 (1945); *C. A.* 39: 5430 (1945).
The results of tests by the Quebec Roads Department on the use of sulphite waste liquor as a stabilizing agent for road foundations are summarized. The solubility of the sulphite liquor is not an objection, because after drying the soil becomes waterproof to underground water. Bearing capacity is increased as well as resistance to shear of a stabilized gravel. At 3% water content, resistance increased 2.5 times for a 2% lignin extract.

Thönnessen, K., Ger. pat. 743,092 (Oct. 28, 1943); *C. A.* 40: 541 (1946).
A binding for casting molds and cores is made of sulphite liquor to which is added 5 to 15% of urea.

Bjornstad, P. L., Norw. pat. 66,648 (Aug. 16, 1943); *C. A.* 40: 1318 (1946).
Ready-sized paper, etc., is treated with fillers and (or) pigments suspended in sulphite or sulphate waste liquor. Preferably, the waste liquor is neutralized with milk of lime and admixed with another binding agent, such as casein, size, glue, dextrin or starch. In a modification, the sulphite waste liquor is concentrated and then diluted with a nonaqueous solvent, e.g., methyl or ethyl alcohol in which dyes may be dissolved.

Bodnár, L., *Magyar Kém. Lapja* 2: 245–251 (1947); *C. A.* 41: 7708 (1947).
Sulphite waste liquors may be used in emergency cases to replace molasses as a binding material in briquetting charcoal powder in the air-dry state. The briquets must be heated to 160° to remove the volatile components of the sulphite waste liquor.

Schwabe, K., *Holzforschung* 2: 24–28 (1948); *C. A.* 42: 3954 (1948).
Bricks molded from diatomaceous earth with fermented spruce sulphite waste liquor developed cracks on drying and baking which rendered the product useless. The formation of these

cracks could be avoided if drying proceeded very slowly at low temperatures (not feasible economically) or when nonfermented spruce or uninoculated beech sulphite waste liquors were employed. No cracks appeared when molasses was added to the fermented liquor. The cracks which develop when fermented liquor alone is used as a binder in molded bodies were found to be due to relatively rapid evaporation. Surface skin formation apparently delays evaporation at the later stages.

PHELPS, G. W., *Ceram. Age* 53: 11–12, 40 (1949); *C. A.* 43: 2380 (1949).
The use of clay as a base for linoleum cements containing sulphite waste liquor, as a filler in the manufacture of paper, as a base coat on wallpapers, and as a base in cold-water paints is discussed.

WILLIAMS, R. O., U. S. pat. 2,475,766 (July 12, 1949); *C. A.* 43: 7686 (1949).
Waste paper consisting of 70% nonabsorbent, 15% absorbent, and 15% semiabsorbent paper is torn in a nodulator into relatively small nodules. Binders (10%) such as aqueous sodium silicate, concentrated sulphite waste liquor, emulsified rosin, or pitch are mixed with the paper. The mixture is molded into logs or briquet-shaped pieces.

3. TANNING MATERIALS

WILSON, J. A., AND PORTH, I. H., *J. Am. Leather Chem. Assoc.* 38: 20–30 (1943); *C. A.* 37: 5885 (1943).
The fixation of quebracho and ligninsulphonic acid by hide powder and the effects of these agents on the shrinkage temperature of calfskin were studied over the pH range 2 to 8. The results confirm the authors' theory that a true tanning agent must be capable of linking together at least two points in the protein structure. The polyfunctional quebracho is capable of multiple linkage, it raises the shrinkage temperature and is thus a true tanning agent. Ligninsulphonic acid, however, is assumed to combine with the protein by only one group, it does not raise the shrinkage temperature, and is thus not a true tanning agent. The shrinkage temperature is the most reliable criterion of tannage and bears no direct relation to the amount of chemical combination.

VORSATZ, F., U. S. pat. 2,317,832 (Apr. 27, 1943); *C. A.* 37: 6130 (1943).
Conversion products suitable for use in tanning and for other purposes are obtained by heating sulphite waste liquor with water-insoluble organic compounds containing halogen atoms attached to alkyl groups, such as alkyl chloride, ethylene chloride, or benzyl chloride in the presence of sufficient alkali to neutralize the liquor and bind the halogen. The conversion products are recovered from the reaction mixture by acidification.

RIECHE, A., AND RUDOLPH, W., Ger. pat. 726,076 (Aug. 20, 1942); *C. A.* 37: 6490 (1943).
Sulphite liquor from beechwood is fractionated to yield lignin-sulphonates, which are condensed with a mixture of phenols and aldehydes or ketones or phenol alcohols obtained from them. The condensation is carried out in such a manner that the products (suitable for use in tanning) are water-soluble.

NOERR, H., MAUTHE, G., AND MEISTER, M., Ger. pat. 734,473 (Mar. 18, 1943); *C. A.* 38: 1389 (1944).
Tanning agents are prepared by condensing sulphite waste liquor with polyhydric phenols. The latter are prepared by treating with alkali aromatic compounds having two or more substituents that can be changed to hydroxyl. They are mixed with sulphite waste liquor and, if necessary, the mixture is warmed. Either before or after the mixing, the crude phenols are treated with a substance that effects nuclear binding.

GULDEN, P., Ger. pat. 710,752 (Aug. 14, 1941); *C. A.* 38: 1662 (1944).
A tanning agent is described which consists of a mixture of lime, sulphite waste liquor freed of iron, vegetable tanning solutions, and skimmed milk.

GUSTAVSON, K. H., *Tek, Tid. Uppl. C. Kemi* 73: 59–64 (1943); *C. A.* 38: 5427 (1944).
Ligninsulphonic acids are absorbed irreversibly by hide substance and thus fulfill the first fundamental requirement for a tanning agent. The other properties required from a tannin are lacking in lignin derivatives. They do not produce the required degree of improved hydrothermal stability (increased shrinkage temperature and decreased hydrothermal peptization) of the treated hides but reduce the heat stability of the hide in a moist state. Ligninsulphonic acids also diminish the tryptic resistance of the hide considerably. A hide treated with ligninsulphonic acid, upon drying, does not give a leather but a hard transparent sheet. Ligninsulphonic acid in sulphite waste liquor has various molecular weights. The high molecular α- and the low molecular β-fractions can be separated by dialysis or by precipitation with certain aromatic bases. The α-ligninsulphonic acids are isolated as their barium salts and are liberated with sulphuric acid. Both fractions have the same equivalent bond to the protein, but the weight of combined α-ligninsulphonic acid is twice as great as that of the β-acid. The influence of both acids on the shrinkage temperature is the same. Ligninsulphonic acids and lignin extracts cannot be considered as tanning agents from the scientific point of view. Lignin extracts are valuable fillers for use with vegetable tannins and are diluents of natural tannins for the after tanning of heavy hides. The future use of sulphite waste liquor in the leather industry lies in the preparation of lignin derivatives, which, by coupling with ether compounds, will improve the tanning properties of the lignin derivatives.

I. G. FARBENIND, A.-G., Ger. pat. 739,152 (Aug. 5, 1943); *C. A.* 39: 435 (1945).
In order to prepare tanning substances from sulphite waste liquor, resinous, not fully condensed, condensation products with properties of phlobaphene are caused to act on sulphite liquor at an elevated temperature. From this process are exempted condensation products obtained by treating aromatic hydroxy compounds with sulphur and alkalis or with sulphur chloride and those obtained by the action of aldehydes on dihydroxy diaryl sulphones.

NAUTHE, G., MEISTER, M., AND NOERR, H., Ger. pat. 740,269 (Aug. 26, 1943); *C. A.* 39: 435 (1945).
Alkali-extracted substances from ligneous materials are used for tanning.

CHEMISCHE FABRIK J. A. BENCKISER G.M.B.H., Belg. pat. 445,324 (May 31, 1942); *C. A.* 39: 620 (1945).
Highly concentrated or dried sulphite waste liquor is heated at 50 to 150° with phenols in the presence of acids (preferably in equimolecular proportions). The material is suitable as a tanning agent.

I. G. FARBENIND. A.-G., Belg. pat. 444,409 (Mar. 31, 1942); *C. A.* 39: 620 (1945).
Concentration of sulphite waste liquor is obtained by treating the unconcentrated liquor with an alkali and then acidifying with inorganic acids. Separation of the organic matter is obtained by addition to the treated liquor of salt or of further quantities of acid. The material can be used for tanning.

ZELLSTOFFFABRIK WALDHOF., Belg. pat. 443,724 (Jan. 31, 1942); *C. A.* 39: 637 (1945).
Ammonium hydroxide is added to sulphite waste liquor to complete neutralization or to give a slight alkaline reaction. The liquor is then evaporated and mixed with sufficient acid to precipitate the calcium. The neutral calcium-free concentrate is used as a tanning extract.

ZELLSTOFFFABRIK WALDHOF., Belg. pat. 446,537 (Aug. 31, 1942); *C. A.* 39: 832 (1945).
Sulphite waste liquor is passed, at a temperature below 100°C., over bark or wood containing tanning principles. Extraction of the tannins is carried out in a battery of tanks, the hottest liquid being brought into contact first with the most nearly spent tannin-containing material and successively through less spent material at approximately 10° intervals, until it reaches the fresh material at about 50°. The liquid drawn from the last tank is purified if necessary and, after addition of adjuvants, it is concentrated to give a liquor or solid extract.

ZELLSTOFFFABRIK WALDHOF., Belg. pat. 446,477 (Aug. 31, 1942); *C. A.* 39: 840 (1945).
Liquid ligninsulphonates are condensed with aldehydes in the presence of condensing agents if desired. The products are used for tanning, fireproofing, or as adhesives.

ROBINSON, E. A., AND BEACH, R., Can. pat. 424,386 (Dec. 12, 1944); *C. A.* 39: 1073 (1945).
A tanning composition is prepared by reaction of a compound of sexivalent chromium with ligninsulphonic acids or formaldehyde condensation products thereof. The amount of chromium compound is such that the trivalent chromium compound produced will not precipitate on the addition of excess alkali to an aqueous solution of the agent.

TOPOROVSKAYA, K. S., AND KONOVALENKO, P. S., Russ. pat. 59,051 (Feb. 28, 1941); *C. A.* 39: 1073 (1945).
A mixture of sulphonated naphthalene and phenol is condensed with an aldehyde, the product heated with sulphite waste liquor, and treated further with an aldehyde to yield a synthetic tanning agent.

GUSTAVSON, K. H., *Ing. Vetenskaps. Akad. Handl.* No. 177 (1944); *C. A.* 39: 4249 (1945).
A study was made of the irreversible fixation of lignin extracts of various types by hide protein as a function of pH. In the alkaline range sodium, barium, and calcium salts were used. Tanning effect was evaluated by measuring shrinkage temperature, resistance to trypsin, capacity for fixing acid, and degree of swelling. The effect of modifying the hide and the lignin extracts was studied. Ligninsulphonic acid, unlike mineral acids, does not swell hide but has a dehydrating action and pre-

vents ionization of basic protein groups. The anion of ligninsulphonic acid has a marked effect, particularly in neutral or slightly alkaline solution. Fixation of ligninsulphonic acid increases as pH decreases; that fixed at pH 4 to 8 contains less sulphur than that fixed from highly acid solutions. Maximum fixation is reached at pH 2 to 3, instead of pH 1 as for mineral acids; this indicates that peptide groups do not take part directly in the fixation. Saturation with ligninsulphonic acid lowers the shrinkage temperature and resistance to trypsin; this effect is moderate for skin of mammalian origin but great for fish skin. The isoelectric point of collagen treated with ligninsulphonic acid is pH 3.2 to 4.2. More equivalents of ligninsulphonic acid than of hydrochloric acid are fixed by collagen at pH 3 to 5, and a heavy fixation occurs above pH 5.5 to 6.0, the isoelectric point of collagen. Irreversible fixation inactivates acid-binding groups, withdrawing them from the equilibrium. New groups from internally compensated links are then formed leading to further fixation of ligninsulphonic acid. If collagen is treated with either syntan or ligninsulphonic acid, it loses its power to fix the other. Vegetable tannins show a marked affinity toward hide saturated with ligninsulphonic acid but the various tannins show widely different values of irreversible fixation. Vegetable retannage increases shrinkage temperature greatly, but the shrinkage temperature of vegetable-tanned leather is lower than otherwise if ligninsulphonic acid has been used either before or after the vegetable tanning. Vegetable-tanned leather fixes only slight amounts of ligninsulphonic acid at pH 4 to 5 but large amounts at pH 1 to 2. Leather tanned with astringent vegetable tannin binds more acid but less ligninsulphonic acid than leather tanned with mild vegetable tannins. Collagen treated with ligninsulphonic acid does not swell in 0.1 *N* hydrochloric acid and is relatively stable to acetic acid. Deaminized collagen fixes somewhat less ligninsulphonic acid than normal collagen. Pretreatment of collagen with a peptizing agent (3 *M* acetic acid), although greatly increasing the amount of vegetable tannin fixed, does not result in a greater fixation of ligninsulphonic acid; this fact possibly indicates that in the primary fixation of the ligninsulphonic acid ionic valency forces are predominating. The irreversible nature of the fixation and especially the dominating effect of the acid anion in the alkaline range as well as the influence of fixation of ligninsulphonic acid on shrinkage temperature can hardly be explained without assumption of secondary reactions between the fixed lignin and the hide protein. About twice as much of a high molecular weight fraction of ligninsulphonic acid was irreversibly fixed as of a low molecular weight fraction but the number of equivalents of each was about the same. A tanning agent must be irreversibly fixed, must stabilize the collagen lattice so that the shrinkage temperature is raised and resistance to enzymes is increased, and must yield a soft, pliable leather. Although ligninsulphonic acid is irreversibly fixed, it lowers the stability of the lattice and so does not meet the requirements for a tanning agent. Lignin extracts are valuable for filling, dispersing, bleaching, and buffering. The lack of tanning power seems to result from structural deficiencies, especially the low content of phenolic groups, and might be corrected by introduction of active groups.

Schirm, E., U. S. pat. 2,365,599 (Dec. 19, 1944); *C. A.* 39: 4509 (1945).
The condensation product of sulphite waste liquor, an aromatic sulphonamide, and an aldehyde makes a good tanning agent. The sulphite waste liquor should be free of iron and calcium and either dry or highly concentrated. Suitable sulphonamides and aldehydes are mentioned. Condensation is effected by heating the reactants at about 100° until the product forms a clear solution in water.

Rudolph, W., Ger. pat. 742,718 (Oct. 21, 1943); *C. A.* 40: 486 (1946).
Aluminum or aluminum ammonium ligninsulphonates or a mixture of these salts with ligninsulphonic acid, produced by treating sulphite waste liquor with neutral calcium salts and then with aluminum sulphate or aluminum ammonium sulphate, are used for tanning.

Seidel, I., Ger. pat. 742,056 (Oct. 7, 1943); *C. A.* 40: 486 (1946).
Sulphite waste liquor is treated under pressure with compounds containing protein-combining groups (hydroxyaryl, thioaryl, urea, or thiourea) in the presence of a carbonyl compound and an acid which has a soluble calcium salt. A carbohydrate, e.g., starch or its degradation product, may be added to the reaction mixture.

Rieche, A., and Rudolph, W., Ger. pat. 745,097 (Dec. 2, 1943); *C. A.* 40: 486 (1946).
The ammonium or alkali salts of ligninsulphonic acid prepared from beechwood sulphite liquor are suitable tanning agents at a pH of 2 to 7.

National Oil Products Co., Brit. pat. 565,627 (Nov. 20, 1944); *C. A.* 40: 5279 (1946).
A tanning agent is produced by the reaction of a sexivalent chromium compound (e.g., a water-soluble dichromate) with ligninsulphonic acid or its formaldehyde condensation product. The amount of chromium added should be below that which would form a precipitate when an excess of alkali is added to the trivalent chromium-lignin products obtained. Thus tanning agents were obtained from sulphite waste liquors, or their solids, sulphuric acid, and sodium dichromate.

Robinson, E. A., and Beach, R. M., U. S. pat. 2,401,373 and 2,401,508 (June 4, 1946); *C. A.* 40: 5279 (1946).
Ligninsulphonic acids obtained from sulphite waste liquors are used to reduce dichromate to produce tanning liquors which contain chromate complexes. These liquors are produced by the slow, cooled addition of acidified sulphite waste liquor to sodium dichromate, followed by aging and pH adjustment. The sodium dichromate should be 12 to 14% of the solids of the sulphite waste liquor, and 30 to 35% sulphuric acid is preferred for acidification. Neutralization to a pH of 3 to 6 with sodium hydroxide is sufficient for pH adjustment. The resultant leather has the stability to heat gelatinization of chrome-tanned leather, but otherwise may be compared to a vegetable-tanned leather. The tannage may either be a pretannage or a rapid single tannage. The second patent is identical except for the use of formaldehyde-condensed sulphite waste liquors.

Richardson, R. E., Kern, J. G., Murray, R. L., and Sudhoff, R. W., *Shoe and Leather Reptr.* 241, No. 11: 26–29 (1946); *C. A.* 40: 6280 (1946).
The report of a visit to I. G. Farbenindustrie A.-G. Hochst am Main. The methods of preparation of Tanigans SK-7, SK, HN, HZ, G, GB, and Extra F are described in detail.

Curtis, F. J., and Fogler, M. F., *Shoe and Leather Reptr.* 241, No. 11: 29–30 (1946); *C. A.* 40: 6281 (1946).
The report of a visit to I. G. Farbenindustrie A.-G. Elberfeld and Leverkusen. The preparations of Tanigan Extra A, Extra B, and Extra E are briefly described.

Baum, M., Salvesen, J. R., and Harmon, C., *J. Am. Leather Chem. Assoc.*, 41: 300–305 (1946); *C. A.* 40: 6860 (1946).
Hide powders chromed and washed at 18, 23, 28, and 33° were used in the analysis of various tanning materials by the A.L.C.A. method. A 20-minute shake at 23 to 25° was used in all cases. Commercial ligninsulphonate preparations (precipitated and evaporated types) showed a marked increase in tannin content with increasing temperature of preparation of the hide powder. Vegetable tannins (hemlock and chestnut extracts showed no significant change. Experiments with hide powder of various particle sizes prepared at constant temperature showed that the ligninsulphonates give higher tannin values with decreasing particle size of hide poweder. Little effect was noted with the vegetable tannins. It was concluded that a definite temperature of chroming, washing, and shaking should be specified in the A.L.C.A. method when used for the analysis of ligninsulphonate tanning materials.

Kolosov, N. S., U.S.S.R. pat. 66,701 (July 31, 1946); *C. A.* 41: 1862 (1947).
A tanning agent is prepared by neutralizing sulphite waste liquor with ammonia and concentrating without previously precipitating calcium sulphate.

Smetkin, A. I., *Legkaya Prom.* 1946, No. 7/8: 25–26; *C. A.* 41: 2266 (1947).
The preparation of syntan AF is described. It is of the phenol-formaldehyde sulphonic acid type and is used in conjunction with sulphite waste liquor.

Müller, O. A., Brit. pat. 571,916 (Sept. 14, 1945); *C. A.* 41: 4327 (1947); U. S. pat. 2,418,981 (Apr. 15, 1947); *C. A.* 41: 4670 (1947).
Tanning materials are prepared by the action of chlorine on waste sulphite liquor which may have been fermented and dealcoholized and freed from free or loosely combined sulphurous acid. The weight of chlorine added is about 10 to 30% of the content of organic substance. After or during the chlorination, the liquor is treated with an alkaline substance until the organically compounded chlorine is eliminated again and a salt of "tannic acid" is precipitated and isolated. Free "tannic acid" is obtained by treating the salt with sulphuric acid.

Buchanan, M. A., Lollar, R. M., and Niemeyer, D. D., *J. Am. Leather Chem. Assoc.* 42: 232–251 (1947); *C. A.* 41: 4941 (1947).
After a careful examination of the literature, the authors came to the conclusion that there were three fundamental variables upon which conclusive evidence was not available, namely, a thorough understanding of the effect of wood type, the effect of cooking conditions, and the type of forces concerned in the reaction between ligninsulphonates and hide substance. As a result of their investigation, they concluded that sulphite waste liquors are not very satisfactory from a leather making

standpoint. They are low-affinity materials and produce empty leathers. However, the species of wood used, and the conditions of sulphite cooking were found to have a definite influence on the leather-making characteristics of the liquors. The best liquors were obtained from spruce when cooked under conditions used to produce rayon pulp, but even these gave a low-grade tannage. The higher molecular weight fractions which were precipitated from solution with high molecular weight amines, for example, β-naphthylamine, gave the best results. Contrary to Gustavson, these authors found that low pH tannages are not necessarily optimal for ligninsulphonates, and that the latter do not always act as disaggregating agents for collagen.

HERFELD, H., *Reichsamt Wirtschaftsausbau, Chem. Ber. Prüf.* Nr. 015 (PB 52017), 843–851 (1942); *C. A.* 41: 4941 (1947).
A discussion of possible tannin substitutes with a reference to the possibility of using sulphite waste liquor.

KUTYANIN, G. I., AND ARBUZOV, G. A., *Legkaya Prom.*, No. 11/12: 40–42 (1946); *C. A.* 41: 5330 (1947).
The stability toward alkali of collagen "tanned" with lignin sulphonic acids was investigated. Increasing the pH of the initial tanning solution decreased the percentage of "irreversibly bound tannin," defined as tannin retained after 20 washings with distilled water. After treatment with excess 0.075 *N* sodium carbonate, the percentage of "tannin" increased with the pH value. These authors concluded that unlike true vegetable tannins, which are, in part, combined reversibly by collagen, all of the ligninsulphonate tannin is bound irreversibly. The absence of a reversibly bound tannin fraction in leather tanned with ligninsulphonate is explained by (1) the weak capacity of ligninsulphonate tannin for colloidal coagulation on the surface of the collagen elements (2) the lack of binding of ligninsulphonic acids with peptide groups by hydrogen bonds owing to the relative scarcity and wide dispersion of phenolic hydroxy groups in ligninsulphonate particles as compared with vegetable tannins.

MAUTHE, G., *Reichsamt Wirtschaftsausbau, Chem. Ber. Prüf.* Nr. 015 (PB 52017), 819–833 (1942); *C. A.* 41: 5742 (1947).
The structure and uses of some of the syntans are reviewed. They are cheaper to transport than vegetable tans and are stable to acids, light, and enzymes. Ligninsulphonic acids can be converted to a tanning material by removing sulpho groups, degrading the molecule, partially freeing phenolic hydroxyl groups, then sulphonating sufficiently to render the product soluble.

BERKMAN, Y. P., *J. Am. Leather Chem. Assoc.* 42: 409–422 (1947); *C. A.*, 41: 7791 (1947).
Conclusions as to the chemical nature and probable mode of preparation of the Tanigans Extra A and D were drawn from analyses and potentiometric curves. The "filling" and "forming" properties were evaluated by determination of the degree of tannage and volume yield. Both products were found to possess properties warranting their designation as replacements for vegetable extracts. Tanigan Extra A has superior "filling" ability and is therefore more suitable for tanning heavy leather. Tanigan Extra B has superior "forming" ability and is therefore more suitable for tanning light feathers.

SCHÜLLER, K., Brit. pat. 590,907 (July 31, 1947); *C. A.* 42: 398 (1948).
Sulphite waste liquor is made alkaline with sodium or potassium carbonate and the ammonia released is boiled off. The liquor is then neutralized with acid and mixed with alcohol to precipitate the inorganic salts. The precipitate is separated from the liquor and the alcohol distilled off; this yields a tanning extract with a purity of 60% or more.

MELANDER, K. H. A., SÄVÖ, G. E., AND JOHANSON, T. I., U. S. pat. 2,445,838 (July 27, 1948); *C. A.* 42: 7530; (1948).
Precipitated calcium ligninsulphonate, obtained by treating sulphite waste liquor with lime, is treated with dry sodium (potassium or ammonium) sulphate, and sufficient sulphur dioxide to reduce the pH to 2 at a temperature below 20°. After the calcium sulphate settles (about 24 hours) the supernatant liquor is removed and evaporated (with steam at 100° and then with air at 30°) to dryness. The presence of sulphur dioxide prevents the sodium ligninsulphonate from darkening. A yield of about 25.5% based on the dry solids in the waste liquor was obtained. The product contained 17% ash, 0.3% calcium, and 7% sodium and is light colored, easily soluble, and suitable for use in tanning and in washing compounds.

HIGHBERGER, J. H., BUMP, A. H., AND O'FLAHERTY, F., *J. Am. Leather Chem. Assoc.* 43: 307–329 (1948); *C. A.* 42: 8506 (1948).
As a result of a laboratory scale investigation of approximately 40 patent disclosures pertaining to the production of synthetic tanning materials, eight were selected for large-scale evaluation. These consisted of an acid condensate of concentrated sulphite waste liquor with resorcinol, and of dispersions in sulphite waste liquor of a dihydroxy-diphenyl sulphone-formaldehyde resin, two modifications of phenol-formaldehyde resins, a blend of a β-naphthalene sulphonic acid-formaldehyde condensate with diphenylolpropane, and an alkaline resorcinol-furfural condensate (in three types of sulphite waste liquor). The products were used in pilot-scale sole-leather tannages, and the resulting leathers were subjected to chemical, physical, and actual wear tests. The results indicated that blends of sulphite waste liquor and an alkaline resorcinol-furfural condensate can produce leather of similar characteristics to those given by natural tannins.

MAEDA, H., *J. Soc. Chem. Ind. Japan*, 45: 267–272 (1942); *C. A.* 43: 1972 (1949).
The tanning mechanism of ligninsulphonic acid was studied by its reaction on gelatin. Coagulation of gelatin dispersed in water was completed by adding ligninsulphonic acid which contained an equivalent amount of sulphonic radical as the free amino radical in gelatin, the latter being ascertained by titrating the solution of gelatin in glacial acetic acid with sulphuric or hydrochloric acid in the same solvent. This shows that the sulphonic radical plays the principal part in bringing the ligninsulphonic acid molecule into contact with the surface molecules of the hide in the tanning process. The importance of the hydroxyl group and the size of the ligninsulphonic acid molecule were also qualitatively verified, the former by comparing the power of coagulating gelatin by ligninsulphonic acid with that of ethyl or acetyl derivatives, and the latter by measuring the softening point of coagulated gelatin by ligninsulphonic acid of various molecular sizes. With the increase in degree of polymerization, the tanning power of gelatin increased. When the sulphite waste liquor was used without treatment, the softening point was low, and its tanning power was weak. This was considered to be due to the presence of a large amount of ligninsulphonic acid of low molecular weight.

DVORSKII, I. M. L., AND ERUKHIMOVICH, M. I., *Legkaya Prom.* 8, No. 10: 25–26 (1948); *C. A.* 43: 6449 (1949).
A description of experiments on the production of tanning materials from sulphite liquor after fermentation to produce alcohol.

JOHNSON, A. M., AND MARSHALL, H. B., *Pulp Paper Mag. Can.* 50, No. 8: 98–104 (1949); *C. A.* 43: 8677 (1949).
A survey of the utilization of sulphite waste liquor as a tanning agent with 206 references.

OSAKEYHTIO, A. A., Norw. pat. 74,547 (Jan. 31, 1949); *C. A.* 44: 3714 (1950).
Sulphite waste liquors or ligninsulphonic acids obtained therefrom are treated with Cl_2 either before or after saponifying the sulphonic acids present by treatment with alkali at raised temperatures. In an example, sulphite waste liquor is chlorinated with 2 mols of Cl_2 at 18° and then heated to 170 to 175° with NaOH. After acidifying to precipitate the product, it may be used as an emulsifier or tanning agent or used as an artificial resin.

Postwar Government Reports of the German Tanning Industry

C.I.O.S. XXIII-25.
The preparations of Tanigans Extra B, A, and E are described; the two latter contained sulphite waste liquor.

FIAT 450.
Hansa tanning agents were described by Prof. K. Freudenberg as being of rather poor quality.

FIAT 962.
The preparation of Tanigans Extra A, C, and F are described. Tanigan A is prepared as follows: 4100 kg. phenol and 1235 kg. sulphuric acid are heated under vacuum and the excess phenol and water distilled off. The pH is then adjusted to 6 to 7 with sodium hydroxide, diluted and treated with formaldehyde and sodium ligninsulphonate (derived from crude calcium-base sulphite liquor). The resin so obtained is precipitated with ammonium sulphate and then with hydrochloric acid. Tanigan C is rather similar but contains cresols and sodium bisulphite. Tanigan F contains no sulphite waste liquor.

BIOS 216.
This report includes a brief account of an interview with K. Freudenberg. In his opinion there was no immediate possibility of the development of a process which would utilize lignin on a large scale. He concluded that the best way to use considerable amounts of lignin would be the development of many processes using limited quantities.

BIOS 240.
A description of the synthetic tanning agents and auxiliary products produced by I. G. Farbenindustrie.

BIOS 762.
This is a detailed account of the manufacture and practical ap-

plication of German synthetic tanning materials and related substances. The function of sulphite liquors in these syntans is primarily as a dispersing agent, although they do react with hide substance to some extent. The following methods for the use of sulphite waste liquor in tanning are included:

(1) As a tanning agent—a dispersion of 100 parts of sulphite waste liquor and 15 parts of sulphonated Novolak (a phenol-formaldehyde condensation product).
(2) As a tanning agent—a mixture of 95% sulphite waste liquor and 5% quebracho.
(3) As a pretan—the hide is exposed to aqueous sulphite liquor the excess of which is removed by pressure. It is then treated with Novolak in acetone solution and finally with water to precipitate the excess Novolak.

It was further reported that a good sole leather could be produced by the use of a mixture of 60% sulphite waste liquor and 40% spruce bark extract. Attempts to improve sulphite waste liquor by treatment with sodium hydroxide were not particularly successful. A mixture of sulphite waste liquor and sodium hydroxide was dried and heated at 100 to 170° with the result that the sulphonate groups were largely removed and the methoxyl converted to hydroxyl. After condensation of this product with a small amount of formaldehyde, the tanning properties were found to have been improved. However, since only poor yields were obtained and many technical difficulties were encountered, the investigation was discontinued.

BIOS 912.
This report is an account of the German sole leather tanneries. In 1944, 50% of the tannin used in Germany was synthetic. Later this figure decreased, but the use of sulphite waste liquor as a filler increased. The synthetic tannins are described with particular reference to the tanigans. Tanigan Extra A was the most widely used.

BIOS 1425.
A lengthy account of the German light leather industry, including a few references to the use of syntans.

BIOS 1538.
A review of some German textile leather producing firms including a few references to the use of syntans.

Balfe, M. P., Redfern, T., Turner, C. G., and Wise, S. G. A., Hobart Publishing Co., New York (1947); *C. A.* 42: 5250 (1948).
The Manufacture and Practical Application of German Synthetic Tanning Materials and Related Substances.

4. FUELS

Pendl, H., Ger. pat. 712,695 (Sept. 25, 1941); *C. A.* 37: 4552 (1943).
Sulphite liquor is concentrated to a 50% solids content and made into a thin unstable emulsion with an oil. The emulsion is evaporated to a 14% water content and used as a fuel (cf. *C. A.* 37: 2162 (1943)).

Pendl, H., Ger. pat. 729,081 (Nov. 12, 1942); *C. A.* 38: 470 (1944).
The preparation of a motor fuel oil from sulphite waste liquor is described. Sulphite waste liquor is evaporated to dryness and the dry material distilled. The oil obtained thereby is used to dissolve fresh evaporated sulphite liquor.

Freeman, H., Can. pat. 434,345 (Apr. 23, 1946); *C. A.* 40: 4521 (1946).
A fuel is prepared by spraying hot sulphite waste liquor over a mass of wet pulp-mill waste woody material to remove the water. The diluted liquor is concentrated by evaporation and again sprayed over the partially dried mass to dry it further, and deposit combustible solids in it.

5. FERTILIZERS

Bollen, W. B., *Pacific Pulp Paper Ind.* 16 No. 12: 30–34 (1942); *C. A.* 37: 3600 (1943).
Experiments are described which show that application of a moderate amount of sulphite waste liquor to the soil increases general microbial activity. Most of the constituents undergo rapid transformation. Sulphite lignin, although resistant like all lignins, is more readily decomposed than many others. The sugars and lignin in sulphite waste liquor stimulate a response on the part of soil microorganisms not unlike that produced by an equivalent amount of organic substance added as straw. As a source of immediately available energy the sugars have potential value for increasing nitrogen fixation by Azotobacter and certain other soil bacteria. Sulphite waste liquor can serve as a sulphur fertilizer. About half of the total sulphur content cannot be considered rapidly available as sulphate, the remainder, existing in sulphonic combination with the lignin, is slowly oxidized. This slow availability would be advantageous, particularly in soils subject to leaching, because it would provide a prolonged more or less constant supply of sulphate in a manner comparable to that in which slowly available humus nitrogen makes possible a small but continual supply of nitrate. Soil reaction is not greatly altered by moderate applications of sulphite waste liquor, the pH is slightly lowered at first but gradually increases to almost normal. No deleterious effect of the waste liquor was observed with applications as high as 60 tons per acre. The fertilizer value of sulphite waste liquor, as evident from the analytical data, is too low to permit general economic application to the soil. However, where situation and facilities permit, sulphite waste liquor might be distributed in moderate concentration in irrigation water as a means of disposing of a nuisance; no toxicity and some increase in soil fertility could be expected from such application.

Scheffer, F., *Bodenkunde u. Pflanzenernähr.* 27: 23–61 (1942); *C. A.* 37: 4174 (1943).
A critical review of the properties of lignin and ligninlike materials and their significance as humus building materials. Lignins are important raw materials for the formation of humins, but not all lignins can be converted into valuable humin materials. There are important differences between genuine lignins and isolated lignins and these latter also differ among themselves in a manner dependent on the method of isolation. It is recommended that measures be taken to improve the reactivity of by-product lignin.

Alderfer, R. B., Gribbins, M. F., and Haley, D. E., *Ind. Eng. Chem.* 36: 272–274 (1944); cf. *C. A.* 35: 6374; *C. A.* 38: 1830 (1944).
An excellent soil structure can be obtained through the judicious use of sulphite liquor. A marked increase is brought about in probable permeability which is in turn brought about by the considerable increase in the number of water-stable granules produced, as shown by aggregate analysis. Addition of an ample quantity of well-balanced fertilizer should precede the sulphite treatment. The mixture should contain 40 to 60 pounds per acre of available nitrogen in order to prevent serious competition between the soil organisms and crop plants for mineral nitrogen. The sulphite liquor contains about 35% lignin, which should contribute to the formation of humus.

Plauson, H., Ger. pat. 738,894 (July 29, 1943); *C. A.* 39: 5391 (1945).
More or less concentrated sulphite liquor, fermented or not, is catalytically (contact catalysts) treated with 10 to 20% nitric acid. The mixture is kept agitated and it is maintained at room temperature or lower. The nitrogen oxides evolved are conveyed into ammonia, lime, or potassium hydroxide solution. The reaction product is neutralized with some neutralizing fertilizer or mineral fertilizer. The product is thickened with gypsum and finally powdered.

Aries, R. S., *Northeastern Wood Utilization Council Bull.* No. 7: 56–62 (1945); *C. A.* 40: 1623 (1946).
A discussion of the possibilities of a coordinated research on the economical and technical aspects of the use of lignin as a fertilizer. If lignin or other pulp-mill wastes prove uneconomical the hydrolysis of sawdust or chipped wood offers other possibilities.

Seiberlich, J., *Northeastern Wood Utilization Council Bull.* No. 7: 49–52 (1945); *C. A.* 40: 1624 (1946).
The fundamentals of lignin chemistry as applied to fertilizers are discussed.

Eweson, E. W., U. S. pat. 2,392,811 (Jan. 15, 1946); *C. A.* 40: 2258 (1946).
In a variation of the Heijkenskjold process (cf. U. S. pat. 1,881,557 (*C. A.* 27: 559)), a hygroscopic fertilizer containing lignin, vitamins, and yeast is made by the propagation of various races of yeast in neutralized sulphite waste liquor and drying the mash at 140 to 200°F. The lignin coats and protects the yeast cells from disintegration until sufficient soil moisture penetrates the coating and permits a balanced utilization of the yeast components by the plant.

Stephenson, R. E., *Pulp and Paper Ind.* 19. No. 12: 68–70 (1945); *C. A.* 40: 4162 (1946).
Experiments are described in which the quantities of sulphite waste liquor, which are safe to use in irrigation water, are determined. It appears from the results that it is safe to use the liquor in irrigation water to the extent of 0.5 inch per acre (60 tons per year). This quantity would add about 800 pounds of sulphur, 200 pounds of calcium, 180 pounds of magnesium, 16 pounds of potassium, 1 ton of sugar and 3 tons of lignin to the soil; of these sulphur is probably the most beneficial.

Sauchelli, V., *Am. Fertilizer* 104, No. 3: 7, 28, 30 (1946); *C. A.* 40: 4834 (1946).
A review of selected investigations on lignin as a fertilizer material and as a soil amendment.

Aries, R. S., *Paper Trade J.* 123, No. 21: 47–51 (1946); *C. A.* 41: 243 (1947).
Calcium ligninsulphonate from sulphite waste liquors increased the yield of shelled beans by 60% when applied at the rate of 10 tons per acre. The use of lignin from dilute acid hydrolysis (Scholler process, cf. *C. A.* 33: 7543) increased the starch content of potatoes by 85%. A partially hydrolyzed wood (containing cellulosic materials), as well as sawdust, gave less satisfactory results. Lignin with surface-active properties, obtained by either acid or alkaline pulping, increases the infiltration rate of water into soil up to a hundredfold; this indicates promise in eliminating erosion and increasing the rain-absorbing capacity of the soil. Applications of sulphite lignin to soil in pots increased the organic content, pH value, soluble phosphorus, and calcium content of the soil. It could be used as a substitute for liming materials and part of the cost written off because of its calcium and magnesium content. The prevention of leaching is an added advantage.

Nikishkina, P. I., *Pedology* (*U.S.S.R.*) 1946, 667–674; *C. A.* 41: 2835 (1947).
Sulphite waste liquor, before and after fermentation to alcohol, ammoniated lignin, cellulose, and humic acid were tested for their capacity for soil structure formation. The sulphite waste liquor and its waste product after fermentation to alcohol increased the absorbed ammonium nitrogen content of the soil, as well as the structural stability of the 3, 2, and 1-mm. aggregates. Ammoniated lignin proved to be the most stable to decomposition.

Aries, R. S., *Northeastern Wood Utilization Council Bull.* 19: 79–120 (1948); *C. A.* 42: 4745 (1948).
A review of the use of lignin as a soil builder.

Sowden, F. J., Atkinson, H., *J. Sci. Agr.* 28: 175–182 (1948); *C. A.* 42: 6976 (1948).
The use of neutralized sulphite waste liquor solids for soil treatment and their effect on crop growth were investigated. On soils where oats responded to applications of nitrogen, response was also obtained by addition of sulphite waste liquor containing nitrogen. Alfalfa responded to the addition of potassium and phosphorus, but not to the addition of nitrogen. Sulphite waste liquor solids had little or no effect on the growth of alfalfa. Sulphur applied as gypsum or with sulphite liquor solids had no beneficial effect on the growth of either oats or alfalfa. The nitrogen was more available in an ammonium-base liquor neutralized with ammonium hydroxide, than in a calcium-base liquor neutralized with ammonium hydroxide.

6. FERMENTATION

Hägglund, E., Heiwinkel, H., and Bergek, T., *J. prakt. Chem.* 162: 2–18 (1943); *C. A.* 37: 3935 (1943).
In the sulphite pulping of wood a part of the dissolved sugar is transformed into an aldonic acid—probably through a sugar bisulphite compound and a labile keto-sulphonic acid. From the sulphite waste liquors obtained with strongly acid bisulphite solution, sugar sulphonic acids can be isolated in small amounts; with less acidic bisulphite solutions, similar acids can be isolated but they differ from those formed with the strongly acid solutions. These sulphonic acids are stable in boiling dilute mineral acids and cold dilute alkalis. Fructose was treated under standard sulphite cooking conditions to form a sulphonic acid derivative which was separated from excess fructose by fermentation of the latter. This sulphonic acid is a new type of sugar derivative. The stable sugar sulphonic acid is not fermentable and appears to have no effect on the fermentation of glucose. In sulphite waste liquor, especially from the manufacture of strong pulps, the sugars may be present as a bisulphite compound. These compounds greatly retard alcoholic fermentation and are themselves not fermentable. The bisulphite compounds are stable within the pH range of 4 to 7; they must be destroyed before fermentation. A sulphite waste liquor which yields 0.64 to 1.0 ml. alcohol per liter, on being neutralized to a pH of 7 to 7.68, gives 6.2 to 7.4 ml. per liter. The presence of sugar sulphonic acids in low molecular weight β-ligninsulphonic acids has been shown experimentally.

Hägglund, E., *Svensk Papperstidn.* 46: 143–148 (1943); cf. *C. A.* 36: 6335; *C. A.* 37: 5233 (1943).
A review of the research activities of the Central Laboratory of the Cellulose Industry during 1942. These activities were devoted largely to problems concerning sugar formation and destruction during the sulphite digestion under varying conditions. Attempts have been made to define the conditions which allow a maximum yield of alcohol, to trace the origin of methyl alcohol in sulphite alcohol and to study the possibility of increasing the alcohol yield and the dry weight of sulphite waste liquor by the addition of waste liquor and wash waters to the cooking acid. Work on the bleaching of pulp has been continued. Other problems are reviewed briefly.

Bergek, T., *Svensk Papperstidn.* 46: 148–152 (1943); *C. A.* 37: 5234 (1943).
The sugars formed during the sulphite digestion react with the bisulphite as long as such is still present in the cooking liquors. Both simple addition products and more stable sulphonic acids are formed. The stability of these products and their effect on the alcohol fermentation are discussed. Owing to the low acidity of the waste liquors from the manufacture of strong pulp, it is difficult to decompose the sugar-sulphite compounds in these liquors by ordinary aeration and neutralization. By increasing the pH of the liquor to above pH 7, these compounds are cleaved and consequently the alcohol yield can now be increased.

Peukert, M. E., *Beiheft Z. Ver. Deut. Chem.* No. 45: *Die Chemie* 55: 60 (1942); *C. A.* 37: 5430 (1943).
A review of biosynthesis in modern industry, including a reference to one of the most important modern investigations, the action of fungi on sulphite waste liquor.

Fink, H., Lechner, R., and Illig, R., *Vorratspflege u. Lebensmittelforsch.* 5: 100–125 (1942); *C. A.* 37: 5501 (1943).
An account of technical investigations into the production of food yeast from sulphite waste liquor of coniferous and deciduous wood. The necessary pretreatment is described. Torula yeast, which can be continuously cultivated, can be used for seeding and does not need added nitrogen in an organic form. Foaming can be overcome by a simple mechanical device without the aid of an antifoaming agent. Sulphite waste liquor from beech and from conifers is compared. The working up of the yeast waste liquor by separation, pressing, and drying, presented no difficulty.

Nepenin, N. N., *Bumazhnaya Prom.* 18, No. 8: 2–12 (1940); *Chem. Zentr.* 1941, I: 3017; *C. A.* 37: 6399 (1943).
The sugar-containing substances present in sulphite waste liquor can be utilized for the production of alcohol, while the residual liquor, which is rich in organic matter, should be utilized in some other manner. A moderate reduction of the calcium content of the cooking acid by a proper cooking procedure should result in an appreciable increase in the sugar content of the waste liquor without reducing the yield or quality of the cellulose. By combining such measures with a rational method of washing, it should be possible to increase the yield of alcohol per ton of cellulose.

Menzinsky, G., *Svensk Papperstidn.* 45: 421–428 (1942); *C. A.* 37: 2573 (1943); *C. A.* 37: 6594 (1943).
A description of a rapid microbiological method for determining the percentage of sugar available for alcoholic fermentation in sulphite waste liquor.

Neumann, F., Ger. pat. 729,842 (Dec. 3, 1942); *C. A.* 38: 456 (1944).
Sulphite waste liquor, especially from beechwood, is adjusted to a pH of approximately 4.3, nutrient salts are added, and the clarified liquor utilized for growing yeast.

Rieche, A., and Hilgetag, G., Ger. pat. 734,471 (Mar. 18, 1943); *C. A.* 38: 1366 (1944).
A purified ligninsulphonic acid is precipitated from sulphite waste liquor which has been previously used for the growth of a protein yeast.

Schmidt, E., *Papier-Fabr.-Wochbl. Papierfabr.*, No. 2: 43–50 (1943); *C. A.* 38: 2200 (1944).
A review of the technical production of beechwood pulp and the preparation of yeast from beechwood sulphite waste liquor.

Appling, J. W., *Paper Mill News* 66, No. 38: 14–15 (1943); *C. A.* 38: 2201 (1944).
A brief discussion of some of the beneficial activities of microorganisms insofar as the paper industry is concerned; yeasts (for fermentation of sulphite waste liquor or of sugars from wood hydrolysis), fungi (for the production of amylase, diastase, and invertase), and acetic, propionic, and lactic acid-producing bacteria.

Rosten, M. M., *Paper Mill News* 66, No. 43: 60, 62 (1943); *C. A.* 38: 2203 (1944).
A brief outline of the production of alcohol from sulphite waste liquor at the Thorold, Ontario, plant of the Ontario Paper Co.

Sankey, C. A., and Rosten, M. M., *Pulp Paper Mag. Can.* 45: 171–178, 188 (1944); *C. A.* 38: 2203 (1944).
A progress report on the development of commercial production of alcohol for war purposes at the Thorold, Ontario, plant of the Ontario Paper Co., using the so-called "reuse of yeast" process developed by Les Usines de Melle (Can. pat. 341,720; 348,549; 402,847).

Soeters, C. J., *Oliëm Vetten Oliezadem* 26: 63–68 (1941); *Chem. Zentr. 1941*, II: 3011; *C. A.* 38: 2226 (1944).
A discussion of the difficulties in the production of fat from various microorganisms on sugar solutions, molasses, and sulphite liquor. The fats are not below vegetable fats in nutritive value and contain palmitic, stearic, oleic, linoleic, and tetracosanic acids.

Peukert, M. E., *Cellulosechem.* 21: 32–34 (1943); *C. A.* 38: 2410 (1944).
Two hypomycetes ("Myzelpilz-biosyn" (I) and (II)) which synthesize protein from sulphite waste liquors and slops have been studied. Of these I acts only in dilute liquors (containing about 1% reducing sugars), whereas II functions very well in liquors containing up to 2.5% sugars. Either I or II can be used in converting slops into protein. The difference between the synthesis effected by I and II on the one hand and *Torula utilis* (III) on the other is discussed. The pretreatments of waste liquors are similar but the nutrient superphosphate requirements for I and II are considerably lower than for III, and the addition of potassium and magnesium salts is unnecessary. The production of the threadlike structures by I and (or) II makes possible a clean-cut separation of protein from the liquor and simplifies washing. The pressed mycelia are stable and palatable and can be used in "vegetable sausage" manufacture. The protein synthesized contains appreciable amounts of diamino groups which are usually lacking in plant protein.

Roschier, H., *Svensk Papperstidn.* 46: 465–471 (1943); *C. A.* 38: 2486 (1944).
Wood chips can be hydrolyzed by heating under pressure with unfermented sulphite waste liquor. Up to 20% of the dry wood substance is converted to reducing substances. By this means, the sugar in sulphite waste liquor is increased to 7 to 8% and steam costs for ethyl alcohol production are much reduced. 70% of the sugars produced can be utilized. The prehydrolysis is best carried out by heating the chips once or repeatedly at 135 to 145°, each time for about 40 minutes. Pulp may also be hydrolyzed by sulphite waste liquor, but dilute acid is better, since the sugars are not decomposed as they are by the high temperatures necessary with sulphite waste liquor. The wood chips are not changed in appearance by hydrolysis with sulphite waste liquor, and are suitable for rayon manufacture and pulping by the sulphate process.

Scheunert, A., *Biedermanns Zentr. Abt. B. Tierernähr.* 13: 329–340 (1941); *Chem. Zentr.* 1941, II: 2834; *C. A.* 37: 3470 (1941); *C. A.* 38: 3694 (1938).
Dried yeast, which had been grown on beechwood sulphite liquor and which had a protein content of about 50%, was found to contain 4 I.U. Vitamin B_1, and 12.5 biological units of the B_2 complex. Feeding experiments on young rats revealed no difference in the food action between this yeast and brewers' yeast. There were no harmful results after feeding rather large amounts of concentrated waste liquor.

Belani, E., *Tech. für Alle* 1943, 342–344; *Chem. Zentr.* 1943, I: 1426; *C. A.* 38: 4749 (1944).
A description of the production of alcohol from sulphite liquor and the methods of dehydration used with special reference to dehydration by gypsum and its advantages.

Lauer, K., *Papier-Fabr.-Wochbl. Papierfabr.* 1943, 258–261; *C. A.* 38: 4798 (1944).
Prehydrolysis of various straws and reeds permits their use in rayon manufacture. The effects of sulphuric acid prehydrolysis of various woods are discussed not only from the point of view of the properties of the resulting pulp, but from the point of view of fermenting the prehydrolyzate in combination with sulphite waste liquor.

Heiwinkel, H., *Svensk Papperstidn.* 47: 265-270 (1944); *C. A.* 38: 5082 (1944).
The sulphite cooking process is accompanied by a considerable decomposition of sugars, partly by oxidation to aldonic acids. Apparently other reaction products are formed which seem to combine with the low molecular weight fraction of the β-lignin sulphonic acids of the waste liquor. Hence the decrease in sugar content is not only caused by the formation of the aldonic acids but also by the formation of products which contain sulphur and exhibit the properties of sulphonic acids. It is very difficult to separate these sulphonic acids from the low molecular weight β-ligninsulphonic acids. Because of this, pure sulphonic acids were prepared from holocellulose and simple sugars (glucose, galactose, and fructose) by pressure heating with sodium bisulphite cooking acid. It is difficult to prepare pure sugar sulphonic acids because of their marked solubility, their instability, and the fact that they could not be separated from the aldonic acids in one operation. The composition of the various sugar sulphonic acids is given. No product containing sulphur could be isolated from lactose and cellobiose.

Pearl, I. A., and Appling, J. W., *Science* 100: 51 (1944); *C. A.* 38: 5253 (1944).
In a study of the growth of *Penicillium notatum* on sulphite waste liquor it was found that lactose may or may not serve as a nutrient for mold growth. It definitely serves as a preservative for the penicillin produced. In parallel experiments with corn steep liquor it was found that starch dextrin can be substituted for lactose to give equivalent yields of penicillin.

Correns, E., *Beih. Z. Ver. deut. Chem. A; Chemie, B: Chem. Tech.* 1943, No. 47: 22–27; *Chem. Zentr.* 1943, I: 2715; *C. A.* 38: 5349 (1944).
Sulphite waste liquor can be used for alcohol production and the fermented spent liquor evaporated or concentrated by diffusion washing.

Hägglund, E., Heiwinkel, H., and Bergek, T., *Cellulosechem.* 21, 108–114 (1943); *C. A.* 38: 5402 (1944).
Sulphite pulping gives rise to 40 to 60% losses of fermentable sugars, the end products being aldonic acids and the intermediate products "sulphites," formed from the simple sugars and sulphurous acid, and sugar sulphonic acids. The sulphites (exemplified by the compound from glucose) have a maximum stability at pH 5 to 7 and retard fermentation markedly, whereas the sulphonic acids are stable over a very wide pH range and have little influence on fermentation. In the case of hard cooks, loosely bound sulphur dioxide increases in amount until 5 to 7 grams per liter of waste liquor is attained. These maximum values correspond approximately to 1 mole sulphur dioxide per mole of glucose. Sugar in the waste liquor continues to add sulphur dioxide as long as inorganic sulphites are present. The sulphites cannot be split readily by aeration as long as the inorganic bisulphites still remain (i.e., as long as the lime has not been fully utilized). The lower the pH, the more complete is the decomposition of the sulphites. On neutralizing the waste liquors from hard cooks inorganic bisulphites are precipitated. Consequently the sugar sulphites are more fully dissociated. On the other hand, the zone of maximum stability for them is reached by this very treatment, and aeration following ordinary neutralization must be protracted. In waste liquors obtained from soft cooks, all inorganic bisulphites have been consumed and the pH is so high that the sulphites are not stabilized and aeration periods may be shortened. Liquors obtained from cooks used in the production of pulp for feedstuffs and rayon give the highest alcohol yields when fermentations are carried out at pH 4.5 to 5. Usually the liquors from hard cooks should be fermented at not less than pH 6. During the fermentation, the pH decreases; this decrease accelerates the decomposition of the sugar sulphites. When, in the case of hard cooks, the pH of the fermentation lies quite near the neutral point, danger of sulphur dioxide evolution is lessened. The sugar sulphites, however, may be largely destroyed by rendering the waste liquors distinctly alkaline. Subsequently the solutions may be acidified and effectively fermented at pH 4.5 to 5. The efficiency of fermentation then approaches that of pure sugar mixtures.

Hellström, N., *Svensk Papperstidn.* 47: 73–74 (1944); *C. A.* 38: 6556 (1944).
Contaminating substances separated in the distillation of sulphite alcohol consisted of acetal and a fraction boiling at 175° containing chiefly *d*-limonene. The acetal could not be purified by repeated distillation.

Hähnel, S., *Svensk Papperstidn.* 47: 75–76 (1944); *C. A.* 38: 6556 (1944).
The removal of terpenes from sulphite alcohol by distillation in a five-column continuous still (of which the first column contained 46 plates) is described.

Groth, B., Eriksson, A., and Holt, T., *Svensk Papperstidn.* 47: 405–414 (1944); *C. A.* 39: 413 (1945).
During the manufacture of sulphite alcohol, several chemical reactions occur in the distillation system. Acetaldehyde formed during the fermentation may combine not only with the alcohols present to form acetals, but also with hydrogen sulphide to form thioacetaldehyde hydrate. Through the catalytic effect of sulphur dioxide, acetaldehyde may be condensed—with simultaneous removal of water—to give compounds of acetal or mercaptal character, which may be further converted to thioparacetaldehyde with varying sulphur content. The formation and reactions of the sulphur compounds have been studied. By decomposition of the thiosulphate present in the fermented liquor, small quantities of hydrogen sulphide are formed simultaneously with sulphur. Because hydrogen sulphide and sulphur dioxide react fairly slowly to form elemental sulphur there is a possibility that the hydrogen sulphide reacts with the aldehydes present in the fermented liquors to form thioacetaldehydes. Methods have been developed for determining the hydrogen sulphide formed during distillation and for detecting minute quantities of thioacetaldehyde compounds in the alcohol.

Roschier, H., and Karppinen, L., *Finnish Paper Timber J.* 25: 40–49 (1943); *Chem. Zentr.* 1943, II: 1930–1931; *C. A.* 39: 413 (1945).
The hemicelluloses of wood can be hydrolyzed advantageously by heating under pressure with unfermented sulphite waste liquor which converts 20 to 22% of the wood into reducing sugars. The residual wood can be pulped by the sulphate but not by the sulphite process. The pulp obtained appears to have normal properties. Hence, it is possible to obtain fer-

mentable sugars as a by-product in sulphate cooking. Curves and tables are given in the original.

Zellstofffabrik Waldhof, Belg. pat. 446,546 (Aug. 31, 1942): *C. A.* 39: 780 (1945).
A process for obtaining yeast from hardwood sulphite waste liquor in which the undiluted liquor, containing 3 to 5% reducing matter (sugars) is added to a fermenting or fermented wort containing fresh leaven. Air is passed through the wort at the rate of 8 to 10 cubic meters per kilogram of dry yeast obtained. By the addition of lime, the liquor is previously brought to a temperature of 60 to 70° and a predetermined acidity (pH 4.2 to 4.4). The fermented wort is drawn off and replaced by fresh diluted wort. The yeast is separated by centrifuging.

Sherrard, E. C., and Kressman, F. W., *Ind. Eng. Chem.* 37: 5–8 (1945); *C. A.* 39: 810 (1945).
A review of processes for the conversion of wood and sulphite waste liquor into ethanol in the United States prior to World War II.

Central Laboratory of the Cellulose Industry, Analysis Committee (Sweden), *Svensk Papperstidn.* 47: 512–515, 552–554 (1944); *C. A.* 39: 1536 (1945).
The text is given of the Swedish Standard CCA II for determining the amount of reducing and fermentable sugar in sulphite waste liquor. The original Schoorl method (*Z. angew. Chem.* 12: 633 (1899)) was suitably modified to make it applicable to the waste liquor. Comparison of results obtained in different laboratories indicated that the agreement was better when using the modified Schoorl method, including that by Kjeldahl-Bertrand.

Lochhead, A. G., and Chase, F. E., *Can. J. Research* 23F: 161–167 (1945); *C. A.* 39: 2177 (1945).
Sulphite waste liquor, boiled and aerated to remove sulphur dioxide served as a medium for growth of certain penicillin and notatin-producing strains of *Penicillium notatum*, after suitable adjustment of reaction and addition of inorganic nitrogen and phosphorus. Without added organic nutrients, or with supplementary sugar only, the antibacterial activity of the culture fluid is due largely to notatin production. A supplement of corn steep liquor has the effect of increasing penicillin and suppressing notatin formation, so that in the presence of added lactose the culture may assay as high as 30 Oxford units of penicillin per ml. Utilizing only the sugar present in the sulphite waste liquor one strain of *P. notatum* produced 3 Oxford units per ml. of culture, while additions of corn steep liquor gave values as high as 11 units, and together with bran, 15 units per ml. Though the results do not suggest the practical use of sulphite waste liquor for penicillin production, where crude culture fluids of highest potency are desired, they suggest the value of further study with microorganisms capable of making use of energy now going to waste for the production of useful metabolic products.

v. Keussler, O., Ger. pat. 744,682 (Nov. 25, 1943); *C. A.* 39: 2842 (1945).
A process for the continuous fermentation of sugar-poor liquids is described, applicable for example to the production of ethyl alcohol from sulphite waste liquor. The spent mash is centrifuged and the yeast suspension thus separated is returned to the first fermenting tub. Within the tubs the yeast is kept in suspension by a circulating pump. The spent mash and yeast it contains is stored in an "after-fermentation" tub, from which the mash is periodically taken to the separator for cleaning.

Central Laboratory of the Cellulose Industry, Analysis Committee, *Svensk Papperstidn.* 48: 134–139 (1945); *C. A.* 39: 3113 (1945).
The text of Swedish method CCA-12 is given. The yield of ethanol in the fermentation of sulphite waste liquor depends on the method of neutralization, the quality of the yeast, and the method of fermentation. The procedures described in this method were selected to conform in principle with mill practice.

Williams, A. E., *Chem. Age (London)* 52: 325–330 (1945); *C. A.* 39: 3113 (1945).
A review of recent developments in the production of industrial alcohol. The relative merits of raw materials as potato stems (haulm), straw, wood (sawdust and meal), peat and sulphite liquor are discussed together with the latest methods to improve conversion, fermentation, efficiency, and recovery. The production of synthetic alcohol is likely to be limited in Great Britain after the war owing to the relative cost and scarcity of raw materials. The future of the alcohol industry, apart from molasses, should be based on the utilization of cellulosic materials accompanying crops which can be grown there. These latter should be converted by biological methods rather than with mineral acids. Although much work has been done on this topic, as yet none of the methods using bacterial cultures found wide commercial application.

Hindsberg, L. E., *Finnish Paper Timber J.* 25: 455–460, 481–488, 504–510 (1943); *Chem. Zentr.* 1944, I: 1248; *C. A.* 39: 3389 (1945).
The problem of obtaining the highest possible alcohol yield at minimum cost is discussed, and the results of practical operation are compared with the theoretical yield. Particular attention is paid to the leaching process from the theoretical standpoint, and to the practical use of successive washings. Laboratory experiments have shown that upward leaching causes channeling in the pulp and that parts of it remain untouched, so that the calculated washing efficiency was only 74%. With leaching in reverse direction the recovery was calculated to be 85%. The experimental results of washing in downward direction are given in a practical example. Two washings gave a recovery of 93 to 94%, with a concentration ratio of 0.83, and an alcohol yield of 113.5 l. per ton of pulp (Enso No. 2.0). The various leaching methods are summarized and critically discussed. The liquor requires 8 to 10 kg. lime for neutralization to above pH 5, and 11 to 12 kg. for practical operation at pH 5.4. Best results in clarification are obtained at 75° and above. The fermentation vat used was provided with so-called effectivators, i.e., wooden cylinders with perforated screens at the open ends, and filled with tree branches. For comparison with the latter, coke, Raschig rings, and chips impregnated with yeast were used. The Raschig rings, and also chips coated with cement, were found to retard fermentation. In laboratory experiments the use of baskets resulted in more rapid fermentation with a saving of 2 to 3 hours. No difference was found in the effect of dead or live yeast. Original materials appear to be the most promising; wood from various trees, except birch, appeared to give the best results. Clean chips apparently have more effect than chips impregnated with yeast; a possible explanation for this is offered. Calculations of production costs are given. The results obtained in practice with the use of a wash liquor tank provided with partitions and of intermediate vats are described. Further details with many diagrams are given in the original.

Bergek, T., *Finnish Paper Timber J.* 25: 373–384 (1943); *Chem. Zentr.* 1944, I: 1248; *C. A.* 39: 3662 (1945).
Previous literature on the effect of various factors on the yield of alcohol from sulphite waste liquor are reviewed, and the possibilities for the more effective utilization of the waste liquors are discussed, such as the use of dilute wash liquors for supplying the acid tower instead of their use, either alone or mixed with water, for preparing the acid. In a study of the sugar compounds formed during sulphite cooking, sugar sulphonic acids have been isolated from holocellulose waste liquors but they were contaminated with aldonic acids. Sugar bisulphite compounds have been synthesized by the method of Kerp. They have a marked stability maximum in the pH range from 5 to 7. At either higher or lower pH, sulphur dioxide is readily split off and the original sugars are obtained again. The sugar sulphonic acids are much more stable, the sulphonic acid groups being split off only very gradually upon treatment with alkali; they seem to have no effect on sugar conversion, whereas the sugar bisulphite compounds markedly retard fermentation. The nature and quantity of sugar-sulphite compounds and other reaction products of unknown constitution, formed upon cooking with either excessive or very small quantities of lime, are discussed. The behavior of liquors from strong pulp, in comparison with those from fodder pulp and rayon pulp, during neutralization and fermentation is explained by the stability of the sugar-sulphite compounds in the various pH ranges. Laboratory experiments have shown that by neutralization of the liquors from a strong pulp with lime to pH 7.4, followed by acidification to pH 4.8 and fermentation, the alcohol yield could be increased to a maximum and the fermentation efficiency raised to 81%. This is approximately the same as can be obtained in laboratory fermentations of sugar or of waste liquors from fodder pulp or rayon pulp. The difficulties encountered in the fermentation of sulphite waste liquors from strong pulps can thus be overcome by a proper method of neutralization. The result of the cooking is believed to depend on a definite minimum of lime in the wood chips rather than on a definite amount of lime in the cooking liquor. Details, with diagrams are given in the original.

Reed, D. L., *Paper Trade J.* 120, No. 25: 68, 70 (1945); *C. A.* 39: 3663 (1945).
The production of alcohol from sulphite waste liquor requires a technology that differs from the processes required for other raw materials, which fact has caused, in part, the lag in its development. Yeast, molds, or bacteria may be useful to convert the sugar, but yeast is probably to be preferred. The production of an edible yeast is feasible with alcohol as a by-product.

Söderström, N. M. G., and Rost, H. F., U. S. pat. 2,381,230 (Aug. 7, 1945); *C. A.* 39: 4717 (1945).
A process for manufacturing yeast from sulphite waste liquor is

described. Depending on the material and the way it is digested, colloidal particles poisonous to yeast may exist in the liquor. The method consists in the usual aeration and reduction of the acidity of the liquor by adding neutralizing agents such as calcium carbonate. The sludge is separated by decantation, centrifugations, or filtration, the latter step so filtering the liquor through a compressed layer or cake which is capable of removing the minute colloidal particles injurious to yeast. The filtering medium is preferably fibrous and of organic origin, for example, cellulose from cotton. Best results are obtained when the liquor is diluted to a sugar concentration of 1.0 to 1.2%. Fermentation is conducted by adding nutrients such as ammonium salts and incubating at 30°. If baker's yeast is to be produced, molasses may be added to the sulphite liquor. A discussion of the surface adsorption forces on the filter bed is presented. Regeneration of the filter after continued use may be effected by boiling in water containing sulphuric acid and an oxidizing agent.

DE FARIA, G., *Rev. quim. ind.* (*Rio de Janeiro*) 14, No. 153: 16–21; No. 154: 18–23 (1945); *C. A.* 39: 5391 (1945).
New sources for the production of ethyl alcohol in Brazil are considered. Sulphite waste liquor, sawdust, and wood waste as a raw material are briefly reviewed.

DANIELSSON, G. J. D., U. S. pat. 2,380,504 (July 31, 1945); *C. A.* 39: 5490 (1945).
Sulphite waste liquor is purified prior to fermentation by treating continuously with a neutralizing agent and then subjecting the neutralized liquor to centrifugal action to remove the sludge. The clarified liquor is suitable for fermentation. The sludge is washed and the recovered sulphite liquor and neutralizing reagent returned to the reagent tank.

TOUSLEY, R. D., *Chem. Met. Eng.* 52, No. 10: 120–123 (1945); *C. A.* 40: 1777 (1946).
From costs of raw materials, processing, and depreciation the following estimates are made: (1) With total cost of pure ethylene at 3 cents per pound, cost of synthetic alcohol would be 13 to 14 cents per gallon. If ethylene is made available at 2 cents per pound, cost of synthetic alcohol probably would be 10 cents per gallon or less. (2) With molasses cost of 5 cents per gallon, cost of molasses alcohol would be about 16 cents per gallon. (3) With corn at 45 cents per bushel, cost of grain alcohol would be 20.5 to 23.4 cents per gallon. (4) For plants of 300 tons of pulp per day, present cost of alcohol from sulphite waste liquor is 15 to 19 cents per gallon of alcohol. Postwar costs would be lower by at least 2 cents per gallon. (5) If waste wood costs $2.00 per ton dry basis f.o.b. processing plant, estimated cost of wood-hydrolysis alcohol is 20.5 to 22.5 cents per gallon.

HELLSTRÖM, N., *Svensk Kem. Tid.* 55: 161–168 (1943); cf. *C. A.* 34: 3429: 38: 6556; *C. A.* 40: 2585 (1946).
A sample of sulphite alcohol fusel oil from commercial production was analyzed directly and after fractional distillation. The components and their relative proportion as obtained from distillation diagrams were: water 17%, ethanol 12%, iso-butanol 12%, iso-amyl alcohol 35%, active amyl alcohol 7%, hexyl alcohol approximately 2%, borneol 6%, guaiacol approximately 0.2%, acids approximately 0.2%, esters approximately 1%. Of the sample components, 92% are herewith accounted for. A maximum of 1 to 2% can be propanol or butanol other than isobutanol; thus propanol is present in sulphite alcohol fusel oil in small amounts only. Fusel oil from fermentation of wood-saccharification sugar (cf. Ender, *C. A.* 32: 3897) contained more active amyl alcohol than as above reported. Hellström believes this is not due to differences in sugars but rather to the fact that the wood saccharification sugars had been fermented with organic nitrogen as nutrient, whereas Hellström's sample was from fermentations where only inorganic nitrogen had been used.

ARNAUDI, C., POLITI, I., AND COLLA, C., *Chimica e industria* (*Italy*) 26: 67–71 (1944); *C. A.* 40: 3223 (1946).
Experiments on the production of yeast from sulphite waste liquor are described. Glucose and mannose contained in sulphite waste liquor are easily fermented. Xylose can be utilized by *Torula utilis* which can live in the presence of 0.1 to 0.6% glucose and at pH 3 to 5. If there is insufficient oxygen present, the production of organic acids increases.

HÖPNER, T., *Cellulosechem.* 22: 72–88 (1944); *C. A.* 40: 3259 (1946).
The loosely combined sulphur dioxide in sulphite waste liquor is partially combined with sugars (particularly glucose) and partially with ligninsulphonic acid. The glucose bisulphite addition compound is stable in acid solution but breaks down with increasing temperature or with increasing pH. According to Hägglund (cf. *C. A.* 38: 5403) it often causes the difficulty encountered in the neutralization of sulphite waste liquor. The behavior of the glucose bisulphite complex and of the loosely combined sulphur dioxide during the neutralization is studied in detail and the results are given in numerous tables and curves. It is found that the equilibrium formation and the equilibrium itself depend on the pH of the solution. With decreasing pH, the velocity of the equilibrium formation decreases and at a pH of 6 to 7 the velocity is so rapid that the combined sulphur dioxide can be titrated as free sulphur dioxide. In order to determine the equilibrium as it exists at a higher pH, the equilibrium is fixed by rapid acidification and the free sulphur dioxide is determined by immediate titration. The organically combined sulphur dioxide is then split off by alkali, the solution again acidified, and the sulphur dioxide rapidly titrated. The behavior of xylose and mannose bisulphite addition compounds is also studied. It is improbable that such sugar bisulphite addition compounds exist in the liquor during the cook but are formed on cooling, with formation of an equilibrium. The results do not indicate whether or not the loosely combined sulphur dioxide is exclusively combined with sugars; they show however that the bisulphite addition compounds do not cause the difficulties experienced in the neutralization of the sulphite waste liquor, and the other somewhat more stable sulphur dioxide complexes, probably of ligninsulphonic acid, are present.

ERICSSON, E. O., *Pulp and Paper Ind.* 19, No. 11: 42, 44, 46, 48 (1945); *C. A.* 40: 4171 (1946).
The sulphite waste liquor used at the Puget Sound Pulp and Timber Co. alcohol plant (2000 gallons per ton of pulp) has a density of 1.05, pH of 2.2, 120 grams per liter total solids, 2 grams per liter free sulphur dioxide, 4 grams per liter loosely combined sulphur dioxide, and 18 grams per liter fermentable sugars. The preparation for fermentation consists in sulphur dioxide removal by stripping with steam, adjustment of acidity to pH 4.5, and cooling to 32°. Fermentation is carried out at 30 to 35° and pH 4.3 to 4.7 for 12 to 20 hours. The yeast is separated by centrifuges. Production is at the rate of 6500 gallons of 190 proof alcohol (20 gallons per ton of pulp).

LOCKE, E. G., *Pulp and Paper Ind.* 20, No. 1: 20–26 (1946); *Paper Mill News* 69, No. 11: 12, 14, 16 (1946); *C. A.* 40: 4172 (1946).
A description of the production of yeast from sulphite waste liquor in Germany. At one time, about 7200 tons of yeast and 6 million gallons of ethyl alcohol per year were produced from sulphite waste liquor. The production of yeast began largely in 1944 and was carried out chiefly in standard plants developed by the Waldhof Co. for producing 1000 tons per year. The process is as follows: sulphite waste liquor is aerated and limed, then cooled and mixed with nutrients. The fermentation is carried out continuously at a pH of 4.5 to 5.0 using an impure strain of yeast. The fermented liquor is sent to a foam breaker and centrifuged, then diluted and centrifuged again, this being the only washing the product receives. It is then concentrated and dried, the method used depending on whether the yeast was required for consumption by humans or animals. Locke points out the possibilities for this process in the Pacific Northwest, a protein-deficient area.

MCCARTHY, J. L., *Pulp and Paper Ind.* 19, No. 11: 35–36 (1945); *C. A.* 40: 4213 (1946).
A brief review is given of the work of Erdtman and Hägglund (*C. A.* 37: 4568). On the basis of 12 pounds total solids per 100 pounds sulphite waste liquor, the estimated composition (in pounds) is ligninsulphonic acid 7.8, fermentable sugar 1.8, nonfermentable sugars 0.6, sugar-sulphur dioxide derivatives and miscellaneous compounds 1.0, and calcium (as calcium oxide) 0.8.

VOITILA, T., *Finnish Paper Timber J.* 26, No. 7A: 36, 38, 40, 42, 44, 46, 48 (1944); *C. A.* 40: 4475 (1946).
In the analysis of alcohol from sulphite waste liquor, the forerun and fusel-oil fractions are separated by distillation, and the main alcohol fraction is analyzed by measurement of density, distillation residue, color, acidity, Barbet permanganate test, and content of esters, aldehydes, furfuryl alcohol, methanol, and sulphur compounds by standard analytical procedures.

SCHÜRCH, A., *Landw. Jahrb. Schweiz*, 59: 880–892 (1945); *C. A.* 40: 4818 (1946).
The favorable effect on the growth of hogs caused by supplementing their diet with yeast from sulphite waste liquor is described.

WINTER, A. O., *Finnish Paper Timber J.* 27: 73–76 (1945); *C. A.* 40: 4879 (1946).
Waste sugars from the wood pulp industries can be used for protein or fat production. The anaerobic growth of yeast or mold on 2 tons of pentoses gives rise to 1 ton of yeast containing 0.5 ton of protein. Similarly, 1 ton of waste sugar would yield 125 to 150 kg. of a fat resembling vegetable oil when used as the nutrient medium in the anaerobic growth of a Fusarium fat mold.

VIRTANEN, A. I., *Finnish Paper Timber J.* 25: 287–290, 292 (1943); *C. A.* 40: 4881 (1946).
Sulphite waste liquor which has been used for the production of

alcohol is treated with ammonium sulphate and superposphate to yield a medium suitable for the growth of yeast.

WALKER, R. D., JR., AND MORGEN, R. A., *Paper Trade J.* 123, No. 6: 43–47 (1946); *C. A.* 40: 5875 (1946).
The sugar fraction of Florida sulphite waste liquor can be utilized in the production of Torula yeast, a high protein material suitable for cattle feed. Pretreatment of liquor with liming is necessary; inoculum from wort agar slants or actively fermenting waste sulphite liquor is suitable. Approximately 90% of the reducing sugars is utilized in a 15 hour-fermentation cycle. Optimum growth conditions for a laboratory scale process in which *Torulopsis utilis* var. *thermophilia* is used are given. The data do not permit conclusions regarding the economics of the process.

TOUSLEY. R. D., *Paper Trade J.* 123, No. 10: 37–40 (1946); *C. A.* 40: 6207 (1946).
A discussion of the economic and market aspects of the production of alcohol from sulphite waste liquor.

HÄHNEL, S., *Tek. Tid.* 72, No. 46: Uppl. A-C, Kemi, 81–86; No. 50: 91–96 (1942); cf. *C. A.* 38: 6556; *C. A.* 40: 6813 (1946).
An investigation of the basic, older, and the newer types of apparatus for the distillation of alcohol from fermented sulphite waste liquor. The calculations of yield, losses, steam consumption, etc., are based on the method of Kirschbaum (*C. A.* 35: 2040).

ARIES, R. S., *Northeastern Wood Utilization Council Bull.* No. 12: 54–75 (1946); *C. A.* 41: 1350 (1947).
Yeasts are important protein and vitamin B suppliers and vary little in analysis, whether of the *Saccharomyces cerevisae* or the *Torula utilis* type. There is little difference whether yeasts are derived from molasses, wood sugars, sulphite waste liquors, or brewer's slops. Nine tables are given comparing the composition of such yeasts with other protein materials, including vitamins B_1, B complex, B_2, and individual amino acids. Wood yeast is an easily digestible food with an energy content of 4600 calories per kilogram and approaches complete animal proteins in nutritional value.

ARIES, R. S., *Northeastern Wood Utilization Council Bull.* No. 12: 76–92 (1946); *C. A.* 41: 1350 (1947).
Yeast from wood sugars and sulphite waste liquor is widely used in Germany as a meat substitute and extender in extracts, soups, sauces, sausages, stuffings, and as a flavoring of vegetable dishes. It is also used for feed as a source of proteins and B vitamins. As a supplement to poor diets, the effect of yeast exceeds that of milk or fish.

ROSTEN, M. M., *Northeastern Wood Utilization Council Bull.* No. 12: 179–189 (1946); *C. A.* 41: 1350 (1947).
The economics of dry food-yeast production from sulphite waste liquor in the United States are discussed. It will cost about 4 cents per pound with a yield of 40 to 50% of reducing sugars. A flow sheet simplifying German practice is proposed.

LOCKE, E. G., SAEMAN, J. F., AND DICKERMAN, G. K., *Northeastern Wood Utilization Council Bull.* No. 12: 95–178 (1946); *C. A.* 41: 1383 (1947).
Report of Joint Objectives Agency, Office of Military Government for Germany (FIAT No. 499). Details are given on the Scholler and Bergius processes. Sulphite waste liquor was the most important source, and *Torula utilis* the most important organism; *Torula pulcherima, Manilia candida, Candida arborea,* and *Oidium lactis* were also used. The "Waldhof" standard plant for sulphite liquor yeast involves recovery and screening of the liquor from blow pits, neutralization and cooling, addition of nutritive salts required for yeast growth, fermenting by a continuous method with aeration and vigorous mixing, recovery of yeast by centrifuging, its concentration and drying. Engineering details and flow sheets are given. The Appendix indicates individual interviews, details on percolation procedures, and translation of chemical engineering reports from the operation of the Mannheim yeast plant.

ARIES, R. S., *Northeastern Wood Utilization Council Bull.* No. 12: 187–193 (1946); *C. A.* 41: 1433 (1947).
Further development is necessary for improving established processes for sulphite waste liquor with simultaneous production of lignin. The ammonium sulphite pulping may provide a cheap yeast nutritent. Research on small Scholler-type wood hydrolysis plants is necessary. The adaptation of feeding data to American conditions will follow the establishment of plants now in construction.

NORD, F. F., AND MULL, R. P., *Advances in Enzymol.* 5: 165–205 (1945); *C. A.* 41: 2530 (1947).
Sulphite waste liquor contains hexoses and pentoses fermentable by yeasts and fusaria to give ethanol. Previous attempts to ferment pentoses in sulphite waste liquor were unsuccessful because of inhibitory substances, but by treating sulphite liquor with lime, *Fusarium lini Bolley* can be grown and ethanol produced.

SCHWIMMER, S., *Cereal Chem.* 24: 70–78 (1947); *C. A.* 41: 2532 (1947).
The concomitant saccharifying action on starch of eight different barley malts in the presence of sweet potatoes, dry sweet-potato juice, sulphite liquor, and the aqueous extract of wheat flour, each as a supplementary source of β-amylase, was determined.

WEIZMANN, C., U. S. pat. 2,415,777 (Feb. 11, 1947); *C. A.* 41: 2854 (1947).
Aliphatic acids can be prepared from sulphite waste liquor by fermenting it with cultures prepared from soils in which beef or carrots have grown. Details of the production of acetic, propionic, and butyric acids are given.

ERICSSON, E. O., *Chem. Eng. Progress* 43, No. 4: *Trans. Am. Inst. Chem. Engrs.* 165–167 (1947); cf. *C. A.* 40: 4171; *C. A.* 41: 3581 (1947).
A description of the plant at the Puget Sound Pulp and Timber Co., Bellingham, Wash., for the production of 6500 gallons of alcohol per day from sulphite waste liquor.

ROSENQUIST, S. O., Swed. pat. 118,141 (Feb. 18, 1947); *C. A.* 41: 6019 (1947).
High-grade alcohol may be made with low steam costs by distillation of a fermented mixture of sulphite waste liquor and molasses. There should be at least 10 cu. m. sulphite waste liquor to 1000 kg. molasses of 50% sugar content.

JOSEPH, H. G., *Sewage Works J.* 19: 60–69 (1947); *C. A.* 41: 6665 (1947).
Sulphite waste liquor contains 12% solids, 2% sugars, and sulphur dioxide both free and combined with lignin. This is passed downward through stripping towers to remove about 25% of the sulphur dioxide; thus it is made less corrosive and more suitable for fermentation. The liquor is stored at 200°F. It is cooled to 90°F., neutralized to pH 5.5 to 6.0, and pumped to the fermenters where yeast is added. After 12 to 14 hours the beer is settled and centrifuged to recover the yeast which is used again in the process. Very little new yeast is ever used. The beer is then distilled, concentrated, and fractionated to remove methyl alcohol (3%), fusel oil, and other impurities. This fermentation reduces the B.O.D. of the original waste 43%.

ADLER, E., *Svensk Papperstidn.* 50, No. 11: 261–266; No. 11B: 9–16 (1947); *C. A.* 41: 7113 (1947).
The loosely combined sulphurous acid causes trouble in the fermentation and evaporation of sulphite waste liquor. Because its origin is not entirely known, the nature of the substances which combine with sulphurous acid was studied. When sulphite waste liquor is neutralized with lime at about 100° to a pH of 6, only part of the loosely bound sulphurous acid is split off. The residual part is split off only after the pH has been raised to above 7. The sulphurous acid is present as an aldehyde bisulphite addition compound. In the search for aldehydes in sulphite waste liquor, it was found that sugar and furfural bisulphite addition compounds play only a minor role in sulphite waste liquor. Glucuronic acid and sugar sulphonic acid combine only in insignificant quantities with the loosely combined sulphurous acid. Hydroxymethylfurfural could not be detected in sulphite waste liquor. Methylglyoxal, formaldehyde, and trioses add sulphurous acid to the extent of almost 100%. The quantity of each in sulphite waste liquor was estimated. There exists a typical difference between sulphite liquors from rayon pulp cooks and hard cooks. In the former, the major part of the loosely combined sulphurous acid is combined with volatile aldehydes, particularly formaldehyde and methylglyoxal, whereas in the latter a considerable part is attached to nonvolatile carbonyl compounds. When sulphite liquor is freed of its loosely combined sulphurous acid and treated with 2,4-dinitrophenylhydrazine, a 2,4-dinitrophenylhydrazone of ligninsulphonic acid, with one dinitrophenylhydrazone group per eight methoxyl groups, is obtained. When the treatment with alkali is omitted, no dinitrophenylhydrazone is formed; this indicates, that in fresh sulphite liquor, the ligninsulphonic acid is present as a bisulphite addition compound.

TYLER, R. G., *Sewage Works J.* 19: 70–74 (1947); *C. A.* 41: 7114 (1947).
The B.O.D. of sulphite waste liquor (35,000 to 45,000 p.p.m.) is due largely to sugars and sulphur dioxide. Alcohol fermentation is anaerobic and due to *Saccharomyces cerevisiae* and the fodder yeast fermentation is aerobic and due to *Torula utilis.* Alcohol fermentation reduced 62% of the sugars and 43% of the B.O.D. in the waste, while fodder yeast production reduced about 68% of the sugars and 32 to 56% of the B.O.D. depending on the efficiency of aeration. These include the B.O.D. removed by stripping the sulphur dioxide before fermentation.

GROTH, B. S., Swed. pat. 118,281 (Mar. 11, 1947); *C. A.* 41: 7650 (1947).
An apparatus for the distillation of alcoholic liquor, particularly fermented sulphite waste liquor is described.

Smirnov, V. A., and Bondarenko, A. N., *J. Applied Chem. (U.S.S.R.)* 20: 97–104 (1947); *C. A.* 41: 7743 (1947).
Sulphite waste liquor, after fermentation of the hexoses, distillation of the alcohol, and utilization of the pentoses for the culture of the yeast *Monilia murmanica*, is usually concentrated through evaporation to 50 to 70% dry matter (mostly ligninsulphonates); of this about 80% are organic substances, 20% inorganic, the amount of reducing matter is about 8 to 9%, pH 5.8 to 6.3. The physicochemical properties (i.e., viscosity, surface tension, foaming ability) of an aqueous solution of this residue at various concentrations have been determined.

Sundman, J., *Finnish Paper Timber J.* 29, No. 4: 52–59 (1947); *C. A.* 41: 7750 (1947).
A fresh sulphite waste liquor contains substantial quantities of sulphur dioxide combined with the sugars present. When the liquor is freed from gas, the sugar bisulphites are almost completely split up giving free sugar. Loosely bound sulphur dioxide in neutralized liquor originates to the extent of 10% from sugar bisulphite and 90% from other aldehyde bisulphites like ligninsulphonic acid. The hexose bisulphites do not influence the degree of fermentation since they decompose during the process to hexose and sulphur dioxide. Neutralization of the sulphite liquor to a pH of more than 7 should be avoided because of the accompanying loss of sugar. At a pH of less than 2, equilibrium between sugar and bisulphite is attained very slowly. A method is suggested for the determination of different sugars on the basis of the degree of dissociation of their bisulphite complexes.

Sundman, J., *Finnish Paper Timber J.* 29, No. 8: 113–117 (1947); *C. A.* 41: 7750 (1947).
A new method has been developed for the determination of different kinds of sugars, based on the dissociation of sugar bisulphites to sugar and bisulphite. The dissociation constants of a number of sugar bisulphites have been determined. Determinations of the sugars in waste sulphite liquor indicate (1) that the hemicellulose of spruce consists mostly of mannans, (2) that sprucewood also contains galactans, (3) that sprucewood does not contain any readily hydrolyzed glucosans.

Forest Products Subcommittee of the Joint Intelligence Objectives Agency, *J. Forestry* 44: 401–410 (1946); *C. A.* 42: 359 (1948).
A review of technical forest products developments in Germany including the improved technology for rapid, continuous fermentation of alcohol or manufacture of protein yeast for food from wood sugar in sulphite waste liquor and from wood-sugar factories.

Saeman, J. F., *Anal. Chem.* 19: 913–915 (1947); *C. A.* 42: 717 *C. A.* (1948).
The Waldhof fermentor used in Germany to ferment sulphite waste liquor for production of food yeast is described. The Waldhof process for fodder yeast production permits fermentation to be carried out continuously at a high rate, maintains a vigorous yeast, and controls foaming. Details are given of changes made by Saeman in the original design used in Germany.

Harris, E. E., Hannan, M. L., and Marquardt, R. R., *Paper Trade J.* 125, No. 22: 34–37 (1947); *C. A.* 42: 718 (1948).
Sulphite waste liquor may be the source of carbohydrates upon which to produce a food yeast for either human subjects or animals. Sulphur dioxide is removed by steam stripping or by precipitation with lime. The resulting solutions are neutralized to a pH of 4 to 4.5, nutrients added, and the solutions inoculated with a yeast of the Torula type. Sulphite waste liquor is sterile and therefore suitable for continuous propagation of yeast. One of the most serious problems is the tendency to excessive foaming. This may be overcome by the use of a mechanical device which allows the gas bubbles to break as the liquid falls down a central draft tube. Four kinds of sulphite liquor were used to propagate yeast:

(1) Sulphite liquor which had been steam stripped to remove sulphur dioxide, neutralized to pH 4.1 with lime, and filtered.
(2) Sulphite liquor neutralized directly with lime to pH 4.1 and filtered.
(3) Sulphite liquor concentrated to 40% total solids.
(4) Magnesium-base sulphite liquor.

The rates of growth of these sulphite liquors have been compared with the rate of growth in sugar solutions produced by the hydrolysis of wood.

McCarthy, J. L., U. S. pat. 2,430,355 (Nov. 4, 1947); *C. A.* 42: 761 (1948).
Sulphite waste liquor is heated to 100° and treated with steam in a countercurrent manner to remove sulphur dioxide and reduce the pH to 3 to 5; it is then suitable for fermentation for the growth of yeast. A sulphite liquor containing 14 grams loosely combined sulphur dioxide (pH 1.5) on steam treatment gave a liquor with 3 grams sulphur dioxide (10 grams sulphur dioxide recovered) and a pH of 4.5. A sulphite liquor with a pH less than 3 may be further neutralized with chemicals.

Tomlinson, G. H., U. S. pat. 2,429,143 (Oct. 14, 1947); *C. A.* 42: 762 (1948).
The residual liquor resulting from the digestion of cellulosic fibrous material is neutralized by the addition of pure magnesium hydroxide to a pH suitable for sugar fermentation. The liquor is then concentrated to above 20% solids, and the fermentable sugars in it are fermented to form ethyl alcohol which is distilled off and rectified. The residue from the distillation is introduced into a high-temperature furnace chamber in which the combustible constituents are burned at a temperature below the fusion temperature of the noncombustible constituents to yield an ash residue containing magnesium hydroxide. The removed ash residue is treated to form fresh magnesium-base sulphite cooking liquor.

Daniels, H. S., and McCarthy, J. L., *Paper Trade J.* 126, No. 5: 43–46 (1948); *C. A.* 42: 2051 (1948).
In preliminary experiments sugars in diluted calcium-base sulphite waste liquor were fermented by *Clostridium polyfermeticum* to yield butyric and acetic acids by batch and continuous flow methods. When the liquor was diluted with an equal volume of water and fermented (10 hours at 30°) in a continuous flow apparatus, about 70% of the original reducing substances were consumed and yields of butyric acid of 40 to 45% (by weight of sugars converted) were obtained. Undiluted sodium-base sulphite waste liquor has also been fermented.

Leonard, R. H., Peterson, W. H., and Johnson, M. J., *Ind. Eng. Chem.* 40: 57–67 (1948); *C. A.* 42: 2051 (1948).
Sulphite waste liquor was prepared for fermentation by steam stripping, followed by precipitation of the sulphite at pH 8.5. After filtration and neutralization with carbon dioxide, the liquor was inoculated with *Lactobacillus pentosus* 124-2. Of several nitrogen-supplying nutrients, malt sprouts were preferable; these were incorporated into a mash with blackstrap molasses and used as the inoculum medium. The fermentation requires 40 to 48 hours at 30° for completion; during this time the pH of the medium is controlled by addition of calcium hydroxide or calcium carbonate. Fermented sulphite liquor was concentrated to 30% of the initial volume, acidified, and extracted with a solvent. Distribution coefficients are given for lactic and acetic acids between water and various solvents; of the 50 tested, 19 were studied with sulphite liquor. Amyl alcohols and isophorone were good solvents. The lactic acid is readily extracted from the concentrate at 90° and the extract washed free of acids with water. The purity of the acid depends on the solvent employed; with amyl alcohol the final aqueous phase after concentration yields lactic acid of 90% strength containing about 8% impurities and 2% water. Sulphite waste liquor (2000 gallons), corresponding to 1 ton of pulp, yields about 285 pounds lactic acid and 75 pounds acetic acid. The cost of raw materials only (no value for sulphite liquor) is estimated at 2 to 3.5 cents per pound of product depending on whether or not the lignin residue is used for fuel.

Arrnaudi, C., Politi, I., and Colla, C., *Chimica e industria (Milan)* 26: 67–71 (1944); *Chimie et industrie* 57: 165 (1947); *C. A.* 42: 2054 (1948).
An outline of the various factors (sugar concentration, yeast content, acidity) involved in the respiration of *Torula utilis* in the presence of glucose. Between 0.1 and 0.6% glucose concentration has little effect. In a medium poor in oxygen increase in the proportion of yeast results in intensification of the fermentative processes and production of organic acids. For pH values between 3 and 5 the acidity has only a slight influence on the intensity of the respiratory process as there is no deficiency of oxygen; in the latter case increase in pH corresponds to a decrease in oxidizing power and an increase in fermenting power. This is attributable to the effect of acidity on the oxidation-reduction potential; this was confirmed by electrometric titration of media at various pH values. *Torula utilis* can utilize xylose; this utilization is the result of characteristics acquired by adaptation to special conditions (lack of other organic compounds) rather than of a constitutional enzyme characteristic. The influence of pH is different from that in the presence of glucose.

Schepp, R., *Das Papier* I: 140–145 (1947); *C. A.* 42: 3564 (1948).
A series of experiments were carried out to determine sulphite cooking conditions which furnish optimum sugar yields. No useful purpose is served in prehydrolyzing wood that is to be converted later into sulphite (rayon) pulp. Prehydrolysis improves neither pulp quality nor the yields of sugar in the waste liquor. The prehydrolysis-sulphite digestion yielded 20% of sugars from spruce and 26% from beech, whereas the single-stage sulphite digestion gave 24 and 30%, respectively (and thus approached far more closely the calculated value of 25.9 and 31.7% total simple sugars). Other disadvantages of the

prehydrolysis are the increased time and steam consumption, and concomitant higher costs. The analyses of the pulps after prehydrolysis and after sulphite cooking are given. By proper control of the sulphite cook, the destruction of sugars can be prevented, which is a marked advantage over the kraft process when by-product conversion becomes of interest.

BURES, J., *Chem. Obzor* 23: 27–29 (1948); *C. A.* 42: 4342 (1948).
Commercial production of ethyl alcohol from sulphite waste liquor is described. Alcohol of 95% concentration can be obtained.

FRIES, K. W., *Lake States Timber Digest* 2, No. 14: 6, 14 (1948); *C. A.* 42: 4748 (1948).
The manufacture of fodder yeast by Torula yeast organisms is discussed.

VOGT, P. H., Swed. pat. 120,794 (Feb. 3, 1948); *C. A.* 42: 7530 (1948)
Sulphite waste liquor to be used for the manufacture of alcohol or other purposes involving evaporation is allowed to stand at a raised temperature, e.g., at 70° before or after partially neutralizing to a pH below 4.5, until the crust-forming calcium sulphate has precipitated, and the neutralization is then taken up to pH 4.5 to 6.5 and thereafter worked up as usual.

TYLER, R. G., AND MASKE, W., *Sewage Works J.* 20: 516–524 (1948); *C. A.* 42: 9070 (1948).
An apparatus for the production of fodder yeast by foam aeration with recirculation of both liquor and foam is described. Reducing sugars were reduced 75%, producing 0.45 gram yeast per gram of sugars used. B.O.D. removals ranged between 35 and 50%. Yeast is separated from the foam drier by centrifuging and is dried in vacuum driers. The process required very delicate adjustment between rates of feed, aeration, return liquor, and return foam. Although cost of production cannot be figured from this apparatus, indications are that the process is feasible.

WURZ, O., *Papier-Fabr.-Wochbl. Papierfabr.* 1944: 356–358; *C. A.* 43: 399 (1949).
ATS in a waste liquor is defined as the ash (A) plus the organic solids (OTS), where A is contingent on the inorganic solids in the water used in the mill and on the calcium oxide content of the cooking liquor. The higher the Bjorkman No. of a pulp, the lower the ATS of the waste liquor, and the higher the ATS per ton of pulp, the higher was the amount of alcohol that could be produced from the waste liquor. Thus a relationship exists between the ATS, degree of pulping, and alcohol yield. Also discussed are the decrease in alcohol formation with increase in calcium oxide content of the cooking liquor, the rate of decomposition of sugar with increasing sugar and sulphurous ion, and relationship between alcohol yield and the length of the cooking periods, the influence of "intensive fermentation" on alcohol yields, and the importance of temperature and pH.

WURZ, O., AND WORSCHA, H., *Papier-Fabr.-Wochbl. Papierfabr.* 1944: 373–375; cf. *C. A.* 42: 7526; *C. A.* 43: 399 (1949).
With neutralization temperatures from 20 to 130°, it was found that the highest alcohol yields were subsequently obtained from those liquors which had been "neutralized" to pH 5.3 at 95 to 100°. Even with raw cooks, such liquors were readily and rapidly clarified.

DUFRENOY, J., AND PRATT, R., *Papeterie* 70: 386–387, 388–391 (1941); cf. *C. A.* 40: 376; *C. A.* 39: 2177; *C. A.* 43: 1972 (1949).
The effect of adding sulphite waste liquor to media used in the production of pencillins was studied in some detail. The possible generic relationship between lignin and penicillin X (4-hydroxybenzyl penicillin) is discussed.

LASSENIUS, T., *Cellulosa och Papper* 398–405 (SPCI 1908–48); *B.I.P.C.* 19: 299; *C. A.* 43: 3969 (1949).
The development of the manufacture of sulphite ethyl alcohol is reviewed.

ENEBO, L., *Svensk Papperstidn.* 52: 101–106 (1949); *C. A.* 43: 4851 (1949).
Small-scale laboratory fermentation tests with sulphite waste liquor were made in which the yields of alcohol and carbon dioxide were determined. The ratio of carbon dioxide to alcohol was 1.01, against 0.957 from the Gay-Lussac formula. The alcohol yield approached 91% of that formula when fermentation took place in evacuated stationary flasks and only 84 to 88% when the evacuated flasks were shaken. It seems that yeast growth in sulphite waste liquor is more extensive than in pure sugar solutions of the same concentration. Various sources for higher carbon dioxide and lower alcohol yield in fermentation of sulphite waste liquor are discussed.

FRIES, K. W., U. S. pat. 2,474,046 (June 21, 1949); *C. A.* 43: 6826 (1949).
Sulphite waste liquor is fermented to lactic acid by strains of *Lactobacillus plantarum*. The sulphite waste liquor is neutralized with lime to pH 8.0 to 8.8 to precipitate free and loosely combined sulphur dioxide, and after removing the precipitated material, the pH is reduced to 6.3 to 6.7 with acid or carbon dioxide, and the temperature is adjusted to 30 to 40°. An inoculum of *L. pentosus* No. 124-2 (or *L. arabinosus*) is added, along with nutrient and the mixture is allowed to ferment about 3 days. Yields of lactic acid are produced equivalent to 70% conversion of the sugar content of the waste liquor. Both the pentoses and the hexoses are converted. The lactic acid can be recovered from the liquor by extraction with isophorone, butyl lactate, or amyl alcohol, or it can be methylated to the methyl ester and recovered as such. Approximately 15,000 pounds of lactic acid can be produced from 100,000 gallons of sulphite waste liquor.

LEDINGHAM, G. A., *Pulp Paper Mag. Can.* 50, No. 7: 93–99 (1949); *C. A.* 43: 7682 (1949).
A review of industrial fermentations of sulphite waste liquor.

KILP, W., *Wochbl. Papierfabr.* 77: 9–12 (1949); *C. A.* 43: 7682 (1949).
A comprehensive and descriptive article on sulphite alcohol and its production.

HAVRANEK, J., *Chem. Zvesti* 1: 322–326 (1947); *C. A.* 43: 8091 (1949).
A discussion of the utilization of sulphite waste liquor by different kinds of fermentation, e.g., butanol-ethanol (*Clostridium butylicum* and *Bacillus macerans*), methylenesuccinic acid (*Aspergillus terreus*), lactic acid (*Lactobacillus pentosus* and *Aspergillus oryzae*) protein (*Torula utilis*), fat (*Endomyces butylicum* and *Rhodotorula*).

ENEBO, L., JOHNSSON, E., AND LUNDIN H., *Svensk Papperstidn.* 50, No. 11B: 72–75 (1947); *C. A.* 43: 8677 (1949).
The effect of concentrating sulphite waste liquor on the rate of fermentation, and yeast and ethanol produced was investigated by bottle tests and semicontinuous lab-scale experiments with sulphite waste liquor from paper pulp as well as from rayon pulp. The fermentation conditions and results are described in detail. Sulphite waste liquor can be concentrated from 30 grams per liter reducing substance (regarded as standard) to 75 grams per liter without appreciably affecting the fermentation efficiency, provided the concentrating operation is carried out by a careful procedure.

ALEXA, G. H., AND SAVEANU, T., *Bull. école polytech. Jassy* 3: 435–442 (1948); *C. A.* 44: 3711 (1950).
The fermentation of a sulphite waste liquor from two Rumanian plants, Letea and Celuloza, gave 1 and 0.8% alcohol by volume, respectively. Attempts to decrease foam during fermentation by sterilizing the neutralized liquor at 80 to 85° for 5 minutes resulted in a 10.9 to 11.5% decrease in yield.

SAMUELSON, H. O., Swed. pat. 125,443 (July 12, 1949); *C. A.* 44: 3670 (1950).
Aldehydes, ketones, hydrogen sulphide, sulphur compounds, and the like are removed from alcoholic liquids, e.g., distillates obtained during the fermentation of sulphite waste liquors, by passing the liquid over an anion-exchange substance containing basic groups, particularly an amine-aldehyde condensation product. In an example, an alcoholic liquid from sulphite waste liquor which contains 15% ethyl alcohol, 0.15% acetaldehyde, and small amounts of sulphur compounds, is treated with SO_2 until it contains 0.7% by weight, and is then filtered through an artificial resin containing an amino group.

SUNDMAN, J., *Finnish Paper Timber J.* 31: 309 (1949); *C. A.* 44: 4193 (1950).
Two moles of alcohol are lost for every mole of acetaldehyde formed as a by-product of sulphite waste liquor fermentation. Variation of the acidity (pH 4 to 6), amount of yeast (0.5 to 10-gram moist weight per liter), and temperature (25 to 27°) has little effect on the amount of acetaldehyde formed. The amount formed increases with the SO_2 content of the liquor. It is therefore advisable to add the acetaldehyde obtained as distillation forerun from the fermented liquor to subsequent fermentations to bind free SO_2 and inhibit formation of acetaldehyde.

YAMAGUCHI, T., *Science Forest Products* (*Japan*) (*Rinsan Kagaku*) 1: 31–36 (1946); *C. A.* 44: 8595 (1950).
From 100 cu. m. of sulphite waste liquor, after neutralizing with CaO, there was obtained 1 ton of dried yeast by adding 300 kg. of rice bran or its equivalent of an accessory N source, $(NH_4)_2SO_4$ or phosphate.

7. EMULSIFYING AGENTS

NOLL, A., Ger. pat. 714,681 (Nov. 13, 1941); *C. A.* 38: 1822 (1944).
A washing agent is prepared from the powdered, water-free or nearly water-free residue of sulphite waste liquor (free of calcium and iron) by intimately mixing with dry soap.

JACOBI, A., Belg. pat. 447,381 (Oct. 31, 1942); *C. A.* 39: 903 (1945).
Fifty-five parts of sulphite liquor and 40 parts of an alkali hydroxide are emulsified with the aid of 5 parts of triethanolamine

as an emulsifier to form a composition suitable for degreasing and de-oiling metals.

RUDY, H., AND WATZEL, R., U. S. pat. 2,385,586 (Sept. 25, 1945); *C. A.* 40: 769 (1946).
Water-soluble materials having lathering power and other surface-active properties are obtained by the condensation of sulphite waste liquor with phenol in the presence of an acid.

MELANDER, K. H. A., SÄVÖ, G. E., AND JOHANSON, T. I., Swed. pat. 116,040 (Mar. 19, 7 1946); *C. A.* 40: 7626 (1949).
Sodium, potassium, or ammonium ligninsulphonates are made from calcium ligninsulphonate (from sulphite waste liquor) by the addition of sodium, potassium, or ammonium sulphate and an acid in such amounts that the solution has an acid reaction. The solution is then filtered or decanted from insoluble substances and evaporated. Sulphurous acid is a suitable acid, and the pH value is preferably adjusted to 2 to 4. It is desirable to evaporate the solution in the presence of sulphurous acid.

ALSMARK, K. O., Swed. pat. 105,402 (Sept. 1, 1942); *C. A.* 40: 7627 (1946).
Sulphite waste liquor is neutralized, and sodium hydroxide and phosphoric acid added in stoichiometric amounts. The precipitate is separated and the liquid brought to a pH of 9 to 11 and used as a lubricant for metal working.

SOC. POUR L'IND. CHIM. Á BALE, Brit. pat. 584,307 (Jan. 13, 1947); *C. A.* 41: 3268 (1947).
Colloidally dispersed cupric oxychloride is prepared by treating a special form of cupric oxide with cupric chloride at 0 to 10° and stabilizing the dispersion with sulphite waste liquor, methyl cellulose, gelatine, or glue.

BARANOV, V. S., *Neftyanoe Khoz.* 24, No. 9/10: 23–27 (1946); *C. A.* 41: 3945 (1947).
The relative effectiveness of aqueous alkaline suspensions of brown coal, sulphite waste liquor, and sulphite-alcohol distillery wash concentrate as a means of improving the filtration characteristics of drilling mud was studied at various pressure differentials ranging from 0 to 60 atm. The filtration values of mud with the addition of about 20% of each of the above agents at 7 atm. pressure and 30 minutes' time were 8.6, 16.7, and 4.0 cc., respectively, as compared with 18.6 cc. in the control test. However, at higher pressure differentials, the sulphite cellulose extract showed a much improved action approaching that of the sulphite-alcohol wash.

LONGLEY, K. D., U. S. pat. 2,419,783 (Apr. 29, 1947); *C. A.* 41: 5721 (1947).
Sodium ligninsulphonate (50 grams) and stearoyl chloride (50 grams) in 100 grams xylene, refluxed for 2 hours, gave 65 grams of the stearic acid ester of sodium ligninsulphonate, an ivory colored wax which is a useful emulsifier where soap is objectionable and in the cosmetic industry for heavy-bodied emulsions. The oleic acid ester is similarly prepared and may be used where lubrication is important, as in rayon knitting.

SALVESEN, J. R., AND BROWNING, W. C., *Chem. Ind.* 61: 232–234 (1947); *C. A.* 41: 6739 (1947).
Ligninsulphonate dispersants prepared from sulphite waste liquor are useful for suspending carbon black, calcium carbonate, sulphur, and clay in water and find application in rubber compounding, ceramics, and insecticides.

SALVESEN J. R., HARMON, C., AND BROWNING, W. C., U. S. pat. 2,491,832 (Dec. 20, 1949); *C. A.* 44: 2753 (1950).
A superior dispersing agent for aqueous slurries of carbon black, clay, and zinc oxide is prepared from sulphite waste liquor from which most of the carbohydrate components have been removed, such as by fermentation, or by precipitating the calcium ligninsulphonate. The latter is then oxidized in alkaline solution (containing one half to two thirds of its weight of NaOH at a pH of 10.5 to 11.0) at 140 to 170° for 30 to 90 minutes by passing oxygen (or equivalent air) through the charge during the cooking process (25 to 40 grams oxygen per 100 grams calcium ligninsulphonate). The insoluble $CaCO_3$ thus produced is removed, the lime-free solution is treated with H_2SO_4 at 60 to 80° at a pH of 3 to 4 and is then further heated to 80 to 90° to coagulate the organic precipitate which is filtered and washed. This product has a lower average molecular weight than the starting material. It is readily coagulated in the presence of acids and salts, particularly sulphate.

OSAKEYHTIO, A. A., Norw. pat. 74,547 (Jan. 31, 1939); *C. A.* 44: 3714 (1950).
(See under Tanning.)

SALVESEN, J. R., AND HARMON, C., U. S. pat. 2,505,304 (Apr. 25, 1950); *C. A.* 44: 6126 (1950).
A demethylated lignin compound useful as a dispersing agent for clay and as as a component in boiler-water treatment is prepared from an aqueous solution of 250 to 350 grams per liter of sodium ligninsulphonate and 50 to 100 grams per liter of NaOH, by heating under pressure at 265 to 325° for 30 to 120 minutes and neutralizing the free NaOH to a pH of about 10.5. Before drying this solution it should be acidified to a pH of 9.0, since when dried from the more alkaline solution, it will combine with sufficient oxygen in storage to heat up the ignition plant.

8. CHEMICALS

A. Vanillin

BRENECK, H., *Ber.* 75B: 561–565 (1942); *B.I.P.C.* 13: 171; *C. A.* 37: 765 (1943).
When sulphite waste liquor is treated with alkali at high temperatures oxidative decomposition of the ligninsulphonic acid occurs; from the alkaline bicarbonate solution vanillin can be recovered by extraction with suitable solvents. The vanillin yield varies with the temperature, the maximum being obtained at 110°. At 150° side reactions occur and the yield of vanillin is decreased. The actual bisulphite pulping procedure is an important factor in determining the vanillin yield from the resulting waste liquors. A short (hard) cook produces liquors which give larger vanillin yields than those from a long (soft) cook. Black liquor from sulphate cooks has the advantage that no alkali addition is required for the oxidation. Black liquor heated to 102 to 103° under otherwise identical conditions yields approximately the same quantity of vanillin as sulphite waste liquor. Evaporation, on the other hand, gives lower results. The waste liquor from beechwood cooks gives only 25% of the vanillin that can be recovered from sprucewood sulphite waste liquor.

PEARL, I. A., AND LEWIS, H. F., *Ind. Eng. Chem.* 36: 664–669 (1944); *C. A.* 38: 4128 (1944).
The reduction of nitrobenzene by sulphite waste liquor and sodium hydroxide was studied at both atmospheric pressure and above. Variables considered were; time of reaction, temperature, concentration and order of introduction of reactants, and the nature of the sulphite liquor. A typical reaction consisted of heating 350 grams sulphite waste liquor containing 50% solids, 90 grams sodium hydroxide in 90 grams water and 72 grams nitrobenzene under reflux for 8 hours, distilling with steam and isolating nitrobenzene, azoxybenzene and aniline from the distillate and azoxybenzene and sodium-*p*-azobenzene sulphonate from the alkaline residue. Azobenzene was obtained at pressures above atmospheric. Vanillin is a by-product of the simultaneous oxidation of the lignin in sulphite waste liquor. Maximum yields of 67.5% azoxybenzene and 24.4% sodium *p*-azobenzene sulphonate were obtained at atmospheric pressure with a minimum of sulphite liquor and an excess of water, respectively. Maximum yields of 65% azobenzene and 67.5% aniline were obtained at pressures above atmospheric, the former with a minimum of sulphite liquor. High temperatures and excessive dilution gave maximum vanillin yields. Sulphite waste liquor from poplar wood gave vanillin-syringaldehyde ratios of 2.5 to 1. Sulphite waste liquor is indicated as a good substitute for zinc dust in the preparation of a mixture of azoxybenzene and sodium *p*-azobenzene sulphonate for direct conversion to benzidine. Material balances were made on nitrobenzene, lignin, and methoxyl entering the reaction. The reuse of desulphonated lignin was considered. Substitution of sodium hydroxide by sodium carbonate gave only small amounts of aniline and no vanillin.

PEARL, I. A., *J. Org. Chem.* 9: 424–429 (1944); *C. A.* 39: 284 (1945).
In the reduction of nitrobenzene with sulphite waste liquor 25% sodium *p*-azobenzene sulphonate is formed as one of the reduction products. It results from the reduction of nitrobenzene by the lignin component in the sulphite liquor, and sulphonation of the reduction product in an active state by inorganic sulphites, either present in the sulphite liquor or formed by desulphonation of calcium ligninsulphonate by the strong alkali at an elevated temperature. Alkali sulphite solutions do not reduce nitrobenzene and in the presence of dextrose only azoxybenzene is formed. Sulphonation must therefore occur with a nitrobenzene reduction product which is formed at a reduction potential characteristic for lignin.

PEARL, I. A., *J. Org. Chem.* 9: 429–434 (1944); *C. A.* 39: 284 (1945).
2-Hydroxy nitrobenzene, 4-hydroxy nitrobenzene, 2-methyl and 4-methyl nitrobenzene, 2-methoxy nitrobenzene, 2-isopropyl nitrobenzene, 2-phenyl nitrobenzene, and 2-chloro nitrobenzene are reduced by means of spruce sulphite waste liquor. The reaction is carried out by heating 0.6 mole of the nitro compound, 90 grams sodium hydroxide in 100 ml. water and 300 grams sulphte liquor containing 50% solids (MeO, 7.1%) under atmospheric or elevated pressure. The yields of reduction products and vanillin were determined for each nitro compound investigated.

LEWIS, H. F., AND PEARL, I. A., U. S. pat. 2,433,227 (Dec. 23, 1947); cf. Brit. pat. 610,814 (Oct. 21, 1948); *C. A.* 42: 2103 (1948).
Isolated lignin substances are oxidized with mercuric oxide in aqueous alkaline solution and acidified with sulphur dioxide to yield mercury-free compounds such as vanillin, acetovanillone,

vanillic acid, and guaiacol. Examples are given using mercuric acetate, dry mercuric oxide, and hot freshly prepared mercuric oxide. If the reaction mixture is initially acidified with acids other than sulphur dioxide, considerable mercurated products are obtained. Methods of recovery of mercury are described. The modifications and limitations of the process are discussed.

PEARL, I. A., *J. Am. Chem. Soc.* 71: 2196–2200 (1949); *C. A.* 43: 6823 (1949).
The oxidation of basic calcium ligninsulphonate (lignin content, 60%) with hot and cold freshly prepared mercuric oxide, with dry mercuric oxide and with silver oxide is described, and the yields of vanillin, acetovanillone, vanillic acid, and neutral compounds given. Oxidation of basic calcium ligninsulphonate with sodium hydroxide and silver nitrate gives 4-nitroguaiacol. Impure vanillic acid can be purified by precipitation of the other acidic materials with lead acetate. The waxy component of the neutral fraction appears to be eicosanol.

ADLER, E., AND HAGGROTH, S., *Acta Chem. Scand.* 3: 86–87 (1949); *C. A.* 43: 7001 (1949).
A phenylpropane derivative other than coniferaldehyde or its sulphonic acid is responsible for part of the vanillin and acetic acid produced on treatment of lignin or its sulphonic acid with alkali. Coniferaldehyde is readily converted to vanillin and acetic acid with 0.1 *N* sodium hydroxide. Ligninsulphonic acid also yields vanillin and acetic acid with 0.1 *N* sodium hydroxide, the reaction stopping with the disappearance of coniferaldehyde and coniferaldehyde sulphonic acid, as determined colorimetrically. Addition of 24% sodium hydroxide, however, results in renewed production of vanillin and acetic acid.

GARDINER, J. A. F., *Pulp Paper Mag. Can.* 50, No. 9: 102–104 (1949); *C. A.* 43: 8677 (1949).
A review of the oxidation of sulphite waste liquor with 34 references.

KLEINERT, *Th. Monatsh.* 80: 582–584 (1949); *C. A.* 44: 1697 (1950).
Sulphite waste liquor was heated with an excess of caustic lime at 100 to 150° under pressure for 3 hours. The precipitate was filtered and stored (without preliminary drying) in glass jars containing air at room temperature. After a few days increasing amounts of formaldehyde were formed. The same results were obtained with purified ligninsulphonic acid. No appreciable amounts of acetaldehyde or vanillin were formed. Vanillin was formed, however, if the precipitate was first dried. Oxidation of the fresh precipitate with hydrogen peroxide prevented or greatly inhibited later formaldehyde formation.

ONTARIO PAPER CO. LTD., Brit. pat. 607,978 (Sept. 8, 1948); *C. A.* 44: 1706 (1950).
The yield of vanillin from lignin-containing substances is increased by the use of finely dispersed air or oxygen in alkaline solutions with a NaOH concentration of 100 grams per liter at a temperature of 150 to 200°, a pressure of 50–200 p.s.i., a partial pressure of oxygen of less than 20 p.s.i. and a reaction time of less than 4 hours. Under these conditions reduced quantities of other auxiliary oxidizing agents are effective. Thus, air dispersed through a mixture of alcohol plant effluent containing 100 grams per liter of NaOH, at a pressure of 110 p.s.i. and a temperature of 169 to 170° for 3 hours yields 11.5% vanillin based on the lignin content. With 16 grams of $CuSO_4$ as the catalyst, in conjunction with air, the yield is 14.4%. Successive reuse of the sludge from the $CuSO_4$ reaction as the catalyst yields 14.8, 15.6, 14.2, and 13.8% vanillin.

SANKEY, C. A., AND MARSHALL, H. B., U. S. pat. 2,489,200 (Nov. 29, 1949); *C. A.* 44: 1706 (1950).
Vanillin formed by oxidation in alkaline medium of lignin-containing substances is extracted from such medium without acidification by means of tertiary butyl alcohol, which is a highly satisfactory solvent for the sodium salt of vanillin and which, although water-miscible, is immiscible with dilute aqueous alkali (at least 5% or equivalent alkali).

LEWIS, H. F., AND PEARL, I. A., U. S. pat. 2,489,380 (Nov. 29, 1949); *C. A.* 44: 1706 (1950).
5-(Hydroxymercuri) vanillin is prepared by treating lignin (in alkali, kraft, or sulphite waste liquors) with a hot solution of NaOH and HgO. The dried precipitate, a white powder m.p. 235 to 236°, is a paper-mill disinfectant, a slime-control agent, and a fungicide in the leather industry.

PEARL, I. A., U. S. pat. 2,483,559 (Oct. 4, 1949); *C. A.* 44: 1706 (1950).
Vanillic acid is produced through oxidation of calcium ligninsulphonate or other lignin substance by Ag_2O in the presence of NaOH. The Ag_2O is freshly prepared by stirring a solution containing 40 parts NaOH into a solution containing 170 parts $AgNO_3$. The Ag_2O is washed free of nitrates, partially dried by suction, suspended in 500 parts water, and 150 parts solid NaOH is added with vigorous stirring. The mixture is heated to about 60° if the heat of solution is not adequate. To the warm mass is added, with stirring, 50 parts of basic calcium ligninsulphonate containing about 60% lignin. The temperature rises to 100° and the mixture boils, the vapors being retained by a reflux condenser. Stirring is continued until the mixture is cool. The metallic silver is removed by filtration or centrifugation, the solution is acidified with SO_2, H_2SO_4, or HCl and extracted with an organic solvent such as ether or benzene The extract is re-extracted with $NaHCO_3$ solution. This extract is acidified, extracted with an organic solvent, and the solvent evaporated. A yield of 28% crude vanillic acid, based on the lignin content of the ligninsulphonate, is obtained. If preferred, the $AgNO_3$ solution may be added to a mixture of the ligninsulphonate and alkali; in this instance it is necessary that a reducing acid, such as SO_2, be used in the acidification to avoid nitration of the vanillic acid.

FLACHSMANN, E., Swiss. pat. 258,294 (May 2, 1949); *C. A.* 44: 3714 (1950).
Vanillin is prepared by heating nitrobenzene with untreated sulphite waste liquor containing ligninsulphonic acid at a temperature of 165 to 175° in an alkaline medium.

PEARL, I. A., *J. Am. Chem. Soc.* 72: 1427 (1950); *C. A.* 44: 4673 (1950).
The $NaHSO_3$-soluble fraction from a Ag_2O oxidation of a *Torulopsis utilis* fermented sulphite waste liquor contains at least 50% 5-carboxyvanillic acid. The fraction (6.8 grams), extracted with 100 ml. boiling ether, treated with 50 ml. dioxan, and the ether removed, gave 3.4 grams of 5-carboxyvanillic acid.

BRYAN, C. C., U. S. pat. 2,506,540 (May 2, 1950); *C. A.* 44: 6624 (1950).
Pure vanillin is obtained by distilling crude vanillin (obtained from lignin) under a moderate vacuum (2 to 15 mm. Hg) at temperatures not exceeding 165° and in the presence of a nonreactive, nonsolvent liquid, aliphatic or chloroaliphatic, saturated hydrocarbon (b.p. 270 to 300°). Thus, 99% vanillin is recovered, and the colored, flavored, and other impurities are left behind. After distillation vanillin is easily separated from the distilling aid and purified to U.S.P. grade.

PEARL, I. A., *J. Am. Chem. Soc.* 72: 2309 (1950); *C. A.* 44: 8106 (1950).
Sulphite waste liquor (2800 grams) containing 72 grams lignin, 180 grams NaOH, and 314 grams $Cu(OH)_2$, heated 5 hours at 170°, yields vanillin and in addition guaiacol, acetovanillone, vanillic acid, 5-carboxyvanillic acid, 5-carboxyvanillin, dehydrodivanillin, and dehydrodivanillic acid.

MARSHALL, H. B., AND SANKEY, C. A., U. S. pat. 2,516,827 (July 25, 1950); *C. A.* 44: 9674 (1950).
Air or oxygen finely dispersed in an alkaline lignin-containing liquor from paper-pulp manufacture produces vanillin. A specially prepared $CuSO_4$ catalyst increases the yield. The lignin-containing liquor, NaOH, and $CuSO_4$ hydrate are heated for 3 hours at 169 to 170°, 110 p.s.i. pressure, air being constantly dispersed in the liquid. The yield of vanillin is equal to 14.4% of the original lignin content of the liquor. The use of oxygen decreases the pressure, time, and temperature of the reaction.

FLACHSMANN, E., Swiss pat. 266,362 (Apr. 17, 1950); *C. A.* 44: 11096 (1950).
Vanillin is made by the oxidation of unpurified sulphite waste liquor containing about 36% ligninsulphonic acid in two steps: (a) oxidation at 140 to 200° with at least 15 cu. m. oxygen per 100 kg. sulphite waste liquor and then (b) oxidation with nitrobenzene (55 to 70 kg. nitrobenzene per 100 kg. sulphite waste liquor) at 165 to 175° for 2 hours (cf. *C. A.* 44: 3714).

FLACHSMANN, E., Swiss pat. 267,115 (June 1, 1950); *C A.* 44: 11096 (1950).
The reaction mixture from the nitrobenzene oxidation of sulphite waste liquor is extracted with hot water to obtain sodium azobenzene-4-sulphonate.

B. Other Phenolic Compounds

GOODELL, E. G., U. S. pat. 2,334,620 (Nov. 16, 1943); *C. A.* 38: 2821 (1944).
A method is employed for continuously recovering phenolic compounds from granular, predried, whole spent liquor solids. The solids in a loosely powdered condition are rapidly heated to a sufficiently high temperature, primarily to char them and render them friable and nonsticky. The charred solids are then conveyed through a destructive distillation zone at a temperature of 400 to 800°F. to distill them destructively. The vapors evolved in this process are removed to a level higher than the destructive zone so that these vapors will have a temperature of 250 to 900°F. The vapors are condensed, and the tarry portion of the condensate removed. The tar is subjected to fractional distillation and to treatment with an alkali such as sodium hydroxide for isolating phenolic compositions.

ERDTMAN, H., *Svensk Papperstidn.* 47: 155–159 (1944); cf. *C. A.* 33: 165; *C. A.* 38: 3465 (1944).
Sulphite liquor lactone or conidendrin (cf. Holmberg, *C. A.* 14: 3230; Erdtman, *C. A.* 29: 1412; Haworth and Slinger,

C. A. 35: 102) is not formed in the sulphite cooking process but is a constituent of the wood itself (cf. Emde, *C. A.* 29: 2353, 3833, 6757) The occurrence of fungicidal phenols in spruce and pine is of great importance. In pines these phenols are protected by the so-called membrane substances which prohibit the direct extraction of the phenols. The question arises then whether or not such resin fractions as conidendrin are protected by membrane substances in spruce also. It is found that young and old sprucewood contain conidendrin in the sapwood and heartwood and that it is apparently deposited inside membranes which are insoluble in ether but soluble in acetone. Conidendrin, therefore, is not extracted from spruce by ether but by acetone. Sulphite cooks of 14 woods of the *Picea* group (of about 35 known) showed that 7 of them contain conidendrin. Of the 11 *Tsuga* species known, 4 were examined and found to contain conidendrin. Of about 40 *Abies* species known, 7 were examined, and in only one of them, *Abies azonica*, conidendrin was found and this result was uncertain. None was found in the *Pinus*, *Pseudotsuga*, or *Larix* species. From *Podocarpus spicatus*, some conidendrin, which is closely related to matairesinol, was isolated.

Pearl, I. A., *J. Org. Chem.* 10: 219–221 (1945); *C. A.* 39: 4755 (1945).
Following the discovery of conidendrin in western hemlock wood (Brauns, *C. A.* 39: 4221), it was isolated from the sulphite waste liquor from the wood in a liquid-liquid extraction in yields of 1.1 grams per liter of digester-strength liquor. On recrystallization from benzene, the conidendrin melted at 255 to 256°; from water or acetone it melted at 238° and then at 255 to 256°; rapid recrystallization from absolute alcohol gave needles melting at 256° but continued heating of the solution gave a conidendrin melting at 238°C.

F. L. Smidth and Co., A/S, Dan. pat. 61,682 (Dec. 13, 1943); *C. A.* 40: 3902 (1946).
Sulphite waste liquor is mixed with peat, dried, and subjected to distillation at 400–600° under normal pressure. Steam may be introduced during the distillation and alkali hydroxide may be added to the mixture at any stage. The distillate contains combustible gases, alcohols, ketones, oils, and phenols, and the residue after washing with water yields a coke.

Salvesen, J. R., Hossfeld, R. L., and Lovin, R. L., U. S. pat. 2,405,450 (Aug. 6, 1946); *C. A.* 40: 7626 (1946).
Various forms of lignin (ligninsulphonic acid, the partially desulphonated acid, soda lignin, thiolignin, acid or Scholler lignin), heated with butanol in the presence of alkali, give a mixture of catechols, monohydric phenols, and neutral oils. Optimum results are obtained at a pH of 10 or above for about 2 hours; the butanol used is from 25 to 75% of the volume of the aqueous alkaline lignin solution or dispersion. Temperature is an important factor; with a partially desulphonated sulphonic acid, the maximum yield of catechols was obtained at 300° (40.4%), of monohydric phenols at 325° (36.9%), and of neutral oils at 350° (71.2%). The product obtained after the preparation of vanillin (600 ml. containing 10% partially desulphonated ligninsulphonic acid and 60 grams sodium hydroxide) and 400 ml. butanol (saturated with water) heated at 300° for 2 hours, gives 41% catechols, 4.7% monohydric phenols, and 29% neutral oils; at 270° the yields of the three fractions are 26.9, 7, and 15.9%, respectively. A certain quantity of the butanol is lost through conversion into acid and higher alcohols, the loss increasing with the temperature.

Salvesen, J. R., Hossfeld, R. L., and Lovin, R. L., U. S. pat. 2,405,451 (Aug. 6, 1946); *C. A.* 40: 7626 (1946).
The loss in butanol encountered in U. S. pat. 2,405,450 can be avoided by the use of a secondary alcohol such as isopropyl alcohol or secondary butyl alcohol. Sodium ligninsulphonate (93 grams) and sodium hydroxide (84 grams in 600 ml. water), heated with 400 ml. isopropyl alcohol at 270° for 2 hours give 32.6% catechols, 4.3% monohydric phenols, and 5.4% neutral oil; with sec-butyl alcohol (350° for 2 hours), there resulted yields of the three fractions of 14.5, 21.8, and 34.9%, respectively. From 90 to 95% of the unused alcohol may be recovered. Other examples are given.

Brauns, F. E., and Pearl, I. A., U. S. pat. 2,417,346 (Mar. 11, 1947); *C. A.* 41: 3625 (1947).
Lignin materials (including calcium ligninsulphonate) and alkali in the ratio of 2:1 are heated with alcohols (such as butanol, isobutanol, pentanols, benzyl alcohol, or cyclohexanol) which do not substantially raise the pressure of water at a temperature of 300°, to effect a degradation to simpler products. A suggested method is given for the separation of the degraded products into aldehydes, phenols, and neutral products.

Cederquist, K. N., Swed. pat. 118,517 (Apr. 8, 1947); *C. A.* 41: 7753 (1947).
A destructive distillation apparatus is described in which cellulosic material or sulphite waste liquor is heated by direct contact with the combustible gaseous products of the distillation combined with another combustible gas.

McLaughlin, R. R., *Pulp Paper Mag. Can.* 50, No. 7: 91–92 (1949); *C. A.* 43: 7682 (1949).
A review of liquid-liquid extraction of sulphite waste liquor with 13 references.

Lackey, H. B., Moyer, W. W., and Hearon, W. M., *Tappi* 32: 469–471 (1949); *C. A.* 44: 1698 (1950).
Conidendrin can be precipitated from sulphite waste liquor by the addition of any one of a number of organic liquids. Aromatic hydrocarbons, halogenated hydrocarbons, ethers, esters, carbonyl compounds, nitro compounds, and some fatty acids precipitate conidendrin from sulphite waste liquor. Among the best are trichloroethylene, toluene, and methyl isobutyl ketone. Aliphatic hydrocarbons, amines, alcohols, and phenols do not precipitate conidendrin. With trichloroethylene the yield is affected adversely by high pH but is not changed by dilution of the sulphite waste liquor. The ratio of trichloroethylene to sulphite waste liquor is important, the optimum recovery being obtained using a ratio of 2:98. When using several hundred gallons of sulphite waste liquor, 0.5% trichloroethylene was sufficient. The recovery varies from batch to batch of the sulphite waste liquor due to unknown factors. By this method conidendrin can be recovered 98% pure in amounts of about 90% of that obtained by solvent extraction for 8 hours.

Grangaard, D. H., U. S. pat. 2,488,186 (Nov. 15, 1949); *C. A.* 44: 2242 (1950).
Phenolic compounds, particularly catechol, are produced by heating lignin with an amine hydrohalide at 200 to 300° for several hours. Thus, 500 parts aniline hydrochloride and 100 parts acid-precipitated lignin (from sulphite waste liquor) are heated at 225 to 230° for 2 to 3 hours with HCl bubbling through the mixture. The mass is diluted with water, HCl is added, and the mixture is extracted with ether. The extract contains 6 to 7 parts catechol and an equal quantity of material of the protocatechuic type; the aqueous solution contains 40 to 60 parts degraded lignin, insoluble in hot concentrated alkali.

C. Furfuraldehyde

Scholz, P., and Kassner, H., Ger. pat. 726,432 (Sept. 3, 1942); *C. A.* 37: 6279 (1943).
2-Furaldehyde is prepared from sulphite waste liquor by raising the acidity of the liquor to an extent where gel formation is inhibited when the liquor is heated at 150 to 210°. As the 2-furaldehyde is formed, it is removed from the reaction chamber by steam.

Voss, W., and Ringel, J., Ger. pat. 742,147 (Oct. 14, 1943); *C. A.* 40: 466 (1946).
Sulphite waste liquor from deciduous trees is concentrated, and the ligninsulphonic acid or the greater part of it is precipitated with a neutral salt. The precipitate is removed and the filtrate is heated for a short time under pressure without adding mineral acid. Furfural is formed and is recovered by steam distillation. This process is particularly suitable for sulphite liquor from beechwood.

Ogait, A., Ger. pat. 745,663 (Dec. 16, 1943); *C. A.* 40: 1034 (1946).
Sulphite liquor from deciduous woods is vaporized in an acid state, and the vapors are extracted with water-immiscible solvents for acetic acid, furfural, and methanol.

Sevon, J. W., Swed. pat. 117,542 (Nov. 5, 1946); *C. A.* 41: 4610 (1947).
The mash obtained during the manufacture of alcohol from sulphite waste liquor, before removal of the alcohol, is wholly or partly freed of its content of bases and rendered acid. The liquor is then subjected to heating under pressure to form 2-furaldehyde. The contents of the column are then expanded and the gases led to a column where the 2-furaldehyde and alcohol are separated. The elimination of the basic content and acidification of the mash may be effected with sulphur dioxide or by means of a base-exchange substance obtained by the polymerization of sulphite waste liquor.

D. Sulphite Turpentine

Tutihasi, R., and Hanazawa, T., *J. Chem. Soc. Japan* 61: 1041–1047 (1940); *C. A.* 37: 258 (1943).
Sulphite turpentine oil obtained from Todo pines in Sakhalin was fractionated and, after eliminating the low distillate (B_{38}, 80 to 100°) containing *p*-cymene, was further fractionally distilled three times at 178–211°/12 mm. The chief distillate at 207 to 210° (sapon. value 199.6) was saponified with 2 *N* alcoholic potassium hydroxide and the acidic portion was further fractionated at 190–210°/3 mm. into methanol and an acid (about 20% of the acidic portion), which upon recrystallization

from petroleum ether, melts at 58.5°, has d_{50} 1.0238, n_D^{50} 1.4891 and $[\alpha]_D^{32.5}$ 85.84; the formula is $C_{15}H_{24}O_3$. The acid absorbed bromine readily and gave a positive color reaction with Legal's reagent, although it was negative with Schiff's reagent, hence it is a ketone. Its neutralization value was the same as the saponification value which indicates that it is not a lactone. The acid contains 2COOH, 1CO and 1C=C (ascertained from the molecular refraction and from the hydrogen addition value) but no methoxyl and is designated todomaturic acid. A number of derivatives were prepared.

Tumbin, P. A., *Bumazhnaya Prom.* 19, No. 4: 16–20 (1941); *Chem. Zentr.* 1942, I: 2213–2214; *C. A.* 37: 3599 (1943).
The potential turpentine yield is calculated from the resin content of the wood and the turpentine content of the resin. In Russian practice 28% of the turpentine is lost during the storage of the wood or during its transportation from the chipper to the digester. Methods of increasing the yield of turpentine are discussed.

Schmidt, E., and Kahle, F. W., Ger. pat. 727,475 (Oct. 1, 1942); *C. A.* 37: 6460 (1943).
A process is described for the recovery of high-grade cymol from gases distilled off in the sulphite pulp process. The gases or their condensate are cooled to a temperature not below 30°, the condensate and tower liquor being maintained at 30 to 60°. The separated cymol is transferred to another container where it is washed with water. The mixture is allowed to settle out and the cymol in the upper layer is separated.

Collins, T. T., Jr., *Paper Ind. and Paper World*, 27: 537–540, 719–722 (1945); *C. A.* 39: 5509 (1945).
A review on sulphite turpentine with 103 references.

Wurz, O., *Textil-Rundschau* II: 161–162 (1947); *C. A.* 41: 6403 (1947).
The recovery of cymene from sulphite waste liquor is described.

Lewis, J. G., *Paper Trade J.* 126, No. 3: 49–50 (1948); *C. A.* 42: 2116 (1948).
p-Cymene is found in the top (as vapor) and side (as liquid) relief from sulphite digesters. It is recovered from the acid-storage tanks and is aerated to remove sulphur dioxide and neutralized with commercial soda ash. The quantity of *p*-cymene formed depends on the species of pine used, the resin content, seasonal characteristics, and aging of the wood. The average yield is 1 gallon per 10 tons of pulp produced. The properties of 13 fractions of a crude *p*-cymene are given.

Tuchihashi, R., and Hanzawa, T., *J. Soc. Chem. Ind. Japan* 45: 273–279 (1942); cf. *C. A.* 37: 258; *C. A.* 43: 5590 (1949).
The constituents of turpentine oil obtained as a by-product of the sulphite process were examined. The low-boiling fraction consisted principally of *p*-cymene. The high-boiling fraction contained a new sesquiterpene keto acid ($C_{15}H_{24}O_3$) as the methyl ester. The medium boiling parts were distilled and a fraction obtained whose properties and behavior corresponded to 1,2,3,4-tetrahydrocadalene. It is assumed that it was formed by dehydrogenation of sesquiterpenes of the cadinene type during the cooking with sulphite liquor.

E. Active Carbon

Norsk Hydroelektrisk Kvaelstofaktieselskab. Norw. pat. 68,562 (Nov. 6, 1944); *C. A.* 40: 1648 (1946).
Concentrated sulphite waste liquor is heated to separate the organic material (cf. Norw. pat. 58,781; *C. A.* 32: 3152) which is then heated to 160 to 250°, washed with acids, bases, or water and thereafter carbonized. If the organic material is separated at a temperature above 160° the further heating step is unnecessary. The carbon obtained has a low ash, e.g., 0.15%.

F. Inorganic Chemicals

Goodell, E. G., U. S. pat. 2,334,621 (Nov. 16, 1943); *C. A.* 38, 2821 (1944).
Calcium-base sulphite waste liquor is spray-dried in a stream of hot gases to form a finely divided mixed inorganic and organic powder. The powder is then suspended in an oxygen-containing gaseous medium and burned under such conditions and for such a time that the calcium content of the powder is converted into minor proportions of calcium carbonate and by reaction, respectively, with the oxygen content of the gaseous medium, and with the sulphur content of the powder, into major proportions of calcium oxide and calcium sulphate. The calcium and sulphur contents of the liquor are thus recovered as calcium carbonate, calcium oxide, and calcium sulphate.

Sherk, D. L., U. S. pat. 2,354,553 (July 25, 1944); *C. A.* 38: 6561 (1944).
A method of treating sulphite waste liquor is employed to recover basic and sulphur constituents in a concentrated form for direct use in preparing fresh sulphite cooking liquor. The liquor is introduced into an electrodialysis cell possessing a number of compartments separated by permeable membranes. An electric current is passed through the cell from one compartment to another so that in one compartment the basic constituents of the liquor are separated and concentrated in basic hydroxide form and in another compartment sulphur constituents are concentrated as sulphur compounds.

Markham, A. E., Peniston, Q. P., and McCarthy, J. L., *Paper Trade J.* 127, No. 1: 64–68 (1948); *C. A.* 42: 7040 (1948).
Ammonium bisulphite waste liquor solids can be pyrolized to yield ammonia equivalent to about 90% of the nitrogen originally present in the liquor. A temperature of about 600° is necessary to complete the pyrolysis in a reasonable time. Satisfactory recovery can be achieved only by nearly complete gasification of the solids. The gas is combustible but of unknown heating value. The atmosphere around the solids during pyrolysis must be humidified. When the waste liquor is fed as a concentrated solution to the pyrolysis retort, the yield of ammonia is about equal to the yield obtained by the use of the solids. Rapid heating of the solids is desirable but not necessary. Although a considerable part of the sulphur is recoverable by the pyrolysis, it is in a form which is not readily available for reuse. A mechanism for the reactions involved is discussed.

Larsson, E. J., and Jonsson, J. H., Swed. pat. 126,919 (Dec. 13, 1949); *C. A.* 44: 3713 (1950).
The smelt obtained after evaporating and burning alkali sulphite and bisulphite waste liquors is dissolved in water to give a solution containing NaHS and NaOH, and CO_2 is passed in to convert all the NaOH to $NaHCO_3$. Advantageously, any SiO_2 which precipitates is filtered off before the $NaHCO_3$ begins to crystallize. The mixture of NaHS and $NaHCO_3$ obtained is heated to drive off H_2S and leave a solution of Na_2CO_3 which is then treated with more CO_2 until crystals of $NaHCO_3$ are formed.

Larsson, E. J., and Jonsson, J. H., U. S. pat. 2,496,550 (Feb. 7, 1950); *C. A.* 44: 6624 (1950).
(See above.)

9. HYDROGENATION

Freudenberg, K., Lautsch, W., and Piazolo, G., *Ber.* 76B: 486–498 (1943); *C. A.* 37: 6655 (1943).
The work on the hydrogenation of lignin in aqueous alkaline solution with hydrogen under pressure has been continued with the object of finding some means of completely or partially replacing elementary hydrogen by some substance which yields hydrogen. Primary alcohols heated in aqueous alcohol solution at 330 to 360° are quantitatively converted into the salts of the corresponding aliphatic acids with liberation of four atoms of hydrogen (Reid, et al., *C. A.* 33: 1657). This reaction was used for the hydrogenation of lignin and the optimum amounts of water, alcohol, and alkali determined. Neutral hydrogenation products are formed almost exclusively, phenols and phenol carboxylic acids being present only in traces or not at all. Under optimum conditions the carbon in the hydrogenation products almost equals the original carbon in the lignin, indicating that alcohol or its dehydrogenation products do not react with the lignin. The fractions obtained on distillation of the hydrogenation products are described. The process is applicable to ligninsulphonic acid salts. Fermented sulphite waste liquor may be hydrogenated simply by heating with alkali although there is insufficient alcohol to complete the reaction. The deficit of hydrogen can be made up with elementary hydrogen, especially if a catalyst is used. Hydrogenation experiments using sodium formate and the Wolff-Kishner reduction are also described. Model substances, e.g., phenol, isoeugenol, behave as they do on ordinary pressure hydrogenation. In the absence of a catalyst, only plurivalent phenols are amenable to hydrogenation by alcohols in alkaline solution. Attempts to replace the elementary hydrogen, in the process whereby lignin preparations are degraded to ether-soluble products by agents yielding hydrogen showed that this is possible in principle but that the yields are only about half those obtained with elementary hydrogen.

Broche, H., and Müller, H., Ger. pat. 728,801 (Nov. 5, 1942); *C. A.* 38: 1344 (1944).
Sulphite liquor or black liquor pitch is extracted under pressure and at an elevated temperature with tetrahydro-naphthalene-cresol or a similar solvent. The extract is then hydrogenated to yield substances suitable for fuel, motor fuel, and lubricating oils.

Freudenberg, K., Lautsch, W., U. S. pat. 2,390,063 (Dec. 4, 1945); *C. A.* 40: 2985 (1946).
An aqueous alkaline solution or suspension of lignin in wood, ligninsulphonic acid, sulphite waste liquor, or black liquor may be hydrogenated to give a 50% yield of phenols. With catalysts (Ni or Pd), introductory hydrogen pressures of 30 to 120 atm. and temperatures up to 260°, 50 to 60% of the lignin is transformed into ether-soluble derivatives. Phenols are the main product, but cycloparaffins are also produced. At higher

temperatures (up to 420°), the proportion of phenols decreases and the cyclic alcohols increase. Since this process is carried out in aqueous media and can be applied to wood waste and to waste pulping liquors as well as to isolated lignin, it is considered to be more practical than hydrogenation in organic solvents.

STUMPF, W., *Svensk Papperstidn.* 50, No. 11B: 164–166 (1947); *C. A.* 41: 7113 (1947).
Hydrogenation of sulphite waste liquor at 350° is a catalyzed reaction, the wall of the autoclave being the catalyst. The absorption of hydrogen is least with a silver wall, but is considerably better with alloy steels containing less than 10% Ni, Cr, or other metal. In the presence of nickel, chromium, ferrochrome catalysts, considerable hydrogen is absorbed. The course of the reaction is dependent on catalyst, temperature, time, hydrogen pressure, and the concentrations of ligninsulphonic acid and alkali. The reaction products contain neutral compounds, phenols of high and low molecular weights and carboxylic, oxycarboxylic, and unsaturated acids. Results are given in curves, but details of the composition of the product are not given. (Cf. *F.I.A.T.* 1136.)

STUMPF, W., *Angew. Chem.* 61: 400–405 (1949); *C. A.* 44: 837 (1950).
Hydrogenation of aqueous alkaline sulphite liquors at 350° and high hydrogen pressures is catalyzed by the autoclave wall when alloy steels containing nickel and chromium are used with more than about 11% chromium. When the ratio of sodium hydroxide to lignin is about 4 to 5, neutral substances, phenols, and acids are formed simultaneously. The use of less than 10% sodium hydroxide gives mainly neutral substances and more than 10% sodium hydroxide in low-alloy autoclaves gives primarily acids. About $^2/_3$ of the lignins are transformed into distillable liquid products. Experimental data are presented.

10. PLASTICS AND ADHESIVES

A. Plastics

POSPIECH, F. K., Ger. pat. 725,533 (Aug. 13, 1942); *C. A.* 37: 6059 (1943).
Condensation products are prepared from sulphite waste liquor solids by treatment with an aralkyl halide, hydroxyaryl, organic carboxylic anhydride or chloride at 100 to 160° under pressure. No condensation agent is required.

PENN, W. S., *Brit. Plastics* 16: 194–198 (1944); *C. A.* 38: 4063 (1944).
A review of lignin plastics with 34 references.

SCHWARTZ, H., *Pulp Paper Mag. Can.* 45: 675–678 (1944); *C. A.* 38: 5614 (1944).
A literature review of lignin plastics with 70 references.

HARMON, C., U. S. pat. 2,371,136 (Mar. 13, 1945); *C. A.* 39: 3160 (1945).
As an improvement on U. S. pat. 1,848,292 (*C. A.* 26: 2864), a solution of basic calcium ligninsulphonate, prepared according to reissued patent 18,268 (*C. A.* 23: 1270; 26: 1122) in aqueous sodium hydroxide, containing sodium hydroxide equivalent to about half the weight of lignin, is heated for half an hour at 175° or for $1^1/_2$ hours at 160°. This causes 35 to 50% of the lignin to become insoluble in sulphuric acid at pH 3.5 but dispersible in aqueous sodium hydroxide. Into this solution, adjusted to 20 to 28% solids content, CO_2 is passed at 65° until a pH of 10.8 is reached causing the separation of calcium carbonate in crystalline form. The filtered solution is then acidified with 12% hydrochloric acid or 35% sulphuric acid to a pH of 3.5 causing the precipitation of the lignin derivative which, after being heated at 95 to 98°, is filtered and washed. The final sodium ligninsulphonate is a dark brown to black solid, moderately soluble in warm water, fusing at higher temperatures. It is insoluble in most organic solvents. By changing the conditions of alkaline hydrolysis, products of high or low sulphur contents are obtained. Treatment of the product with dilute acids gives the free ligninsulphonic acid. By cation exchange other salts such as barium, iron, and lead salts are obtained. These ligninsulphonates may be used in connection with phenol-formaldehyde or other types of synthetic resins, as base exchangers, or as an ingredient in the paste for lead storage battery plates, and for the preparation of other lignin compounds such as esters and ethers.

RHEINBERGER, E., U. S. pat. 2,400,053 (May 7, 1946); *C. A.* 40: 4252 (1946).
A composition suitable for a glue, varnish, or plastic molding powder is made by treating concentrated sulphite waste liquor with a cyanamide compound at temperatures of 50 to 100°. Thus 30 to 250 grams of calcium cyanamide is mixed with 150 to 300 grams of 30°.Bé waste liquor at 50 to 100°. The liquid gradually sets to a tough gel which may be dried and used as a synthetic resin or mixed with 50 to 200 grams wood meal, 2 to 30 grams montan wax, and coloring materials and then dried to 3 to 5% water content for a molding powder to be pressed at 160 to 180°. The ability of the powder to flow is increased by the addition of nitrates or other easily fusible compounds to the wax and calcium cyanamide prior to mixing with the waste liquor. The reaction may also be carried out at temperatures from 200 to 500° under pressure.

OEL U. CHEMIE WERK A.-G., Brit. pat. 569,973 (June 18, 1945); *C. A.* 41: 6078 (1947).
Condensation products from formaldehyde and aliphatic aldehydes described in Brit. pat. 349,556 (*C. A.* 26: 5430) are heated with sulphite waste liquor in the presence of ammonia until the products are soluble in water at a pH above 7, and then precipitated from solution in the acid range. The precipitated products are thermoplastic masses and may be used with fillers to form molding compositions.

OSAKEYHTIO, A. A., Norw. pat. 74,547 (Jan. 31, 1949); *C. A.* 44: 3714 (1950).
(See under Tanning.)

B. Adhesives

V. ARTUS, F., *Gelatine, Leim, Klebstoffe* 10: 97–99 (1942); *C. A.* 37: 6370 (1943).
Substances which "bleed" out of the sulphite waste liquor when it is used as an adhesive, causing discoloration, can be precipitated by lead acetate. The concentrated liquor can be mixed with lime-casein solutions or with calcium oxide and magnesium oxide. On precipitation of the liquor, concentrated to 35° Bé, with magnesium sulphate, there appears a white supernatant substance, *dextron*, which can replace dextrin in sizings, the tannin impurities acting as antiseptics. Formulas are recommended for the preparation of an adhesive for roofing paper, a dextrinlike glue, a quick-drying adhesive, and leatherized paper.

BECHER JR., C., *Seifensieder-Ztg.* 69: 324–325 (1942); *Chem. Zentr.* 1943, I: 477; *C. A.* 38: 3045 (1944).
A discussion of crude and concentrated sulphite waste liquors. The latter can be used as an adhesive.

BECHER JR., C., *Seifensieder-Ztg.* 69: 355–356 (1942); *Chem. Zentr.* 1943, I: 1024; *C. A.* 38: 3748 (1944).
A report on methods of neutralization, purification, and bleaching of sulphite waste liquors for use in the preparation of adhesive materials.

MICKSCH, K., *Nitrocellulose* 13: 143–145 (1942); *C. A.* 38: 4064 (1944).
A review of the utilization of sulphite waste liquor in adhesives.

BJORNSTADT, P. L., Norw. pat. 66,648 (Aug. 16, 1943); *C. A.* 40: 1318 (1946).
Ready-sized paper, etc., is treated with fillers and (or) pigments suspended in sulphite waste or kraft liquor. Preferably, the waste liquor is neutralized with milk of lime and admixed with another binding agent, such as casein, size, glue, dextrin, or starch. In a modification, the waste liquor is concentrated and then diluted with a non-aqueous solvent, e.g., methanol or ethanol, in which dyes may be dissolved.

HAEFNER, R., *Bautech.* 22: 101–104 (1944); *Chem. Zentr.* 1944, II: 1104; *C. A.* 41: 6688 (1947).
An adhesive or calking material can be produced from fine clay and sulphite waste liquor, which can be used as a substitute for bitumen-containing preparations for the protection of buildings.

11. BASE EXCHANGE MATERIALS

ANDERSEN, C. C., Norw. pat. 62,146 (March 11, 1940); *C. A.* 40: 660 (1946).
Sulphite waste liquor is freed from its base content (e.g., calcium, sodium, or ammonium) by treatment in a known manner with a base-exchange substance capable of substituting hydrogen for the base, and the product is dried and heated to a temperature below 130°. The dried mass is boiled with water to remove water-soluble constituents, and a cation-exchange substance obtained capable of exchanging about 1.16 milliequivalents per gram of substance.
Andersen, C. C., Norw. pat. 63,053 (Dec. 9, 1950), addition to 62,146 above; *C. A.* 40: 660 (1946).
The process of the parent case for the preparation of organic cation-exchange substances from sulphite waste liquor, is modified by removing free sulphuric acid wholly or partly before evaporating the liquor. The sulphuric acid may be removed by neutralization or by precipitation, e.g., with barium compounds. In a further modification, the waste liquor is treated with a cation-exchange substance in the acid form, and then mixed with sulphite waste liquor containing a higher content of combined sulphur dioxide than usual. The latter is obtained by treating ordinary waste sulphite liquor with a base and sulphur dioxide. The mixture obtained by this modification is then treated by the process of the parent case.

Norsk Hydroelektrisk Kvaelstofaktieselskab, Norw. pat. 62,807 (Aug. 12, 1940), addition to Norw. pat. 59,070 (*C. A.* 33: 3940); *C. A.* 40: 660 (1946).

The process of the parent specification is applied to a base-exchange reaction between a salt solution and an acid solution, i.e., the following materials are passed consecutively through the base-exchange material obtained by heating evaporated sulphite waste liquor to 150 to 250°, namely: (a) a solution of a salt having the same cation as the salt to be produced, (b) water, (c) an acid having the same anion as the salt to be produced, (d) water. The whole cycle can be repeated. In an example, sodium nitrate is prepared from sodium chloride and nitric acid.

Norsk Hydroelektrisk Kvaelstofaktieselskab. Norw. pat. 65,334 (Oct. 19, 1942); *C. A.* 40: 660 (1946).

Sulphite waste liquor is evaporated to a concentration of 45% solids and is treated hot with an acid cation-exchange substance to replace the cation in the liquor by hydrogen. The solution is then heated to 100 to 150° in an autoclave until the mass solidifies and is dried below 130° (cf. Norw. pat. 62,146). The product is practically insoluble in water, and is an effective cation-exchange substance whose efficiency is about 0.94 mole equivalent per gram.

Lautsch, W., *Die Chemie* 57: 149–154 (1944); *C. A.* 40: 5915 (1946).

The cation-exchanging properties of various lignin resins containing sulphonic acid or carboxyl groups are tabulated. Cation-binding values up to 2 grams equivalents per 1000 grams of dry resins are obtainable. Active anion-exchangers are obtainable by condensing alkali lignin and poly-ethyleneimine with di (chloroethyl) methylamine. Halogen-substituted lignins are also active anion-exchangers and the introduction of amino groups increases the neutral-salt-splitting ability to as high as 3.7 gram equivalents per 1000 grams of exchanger (for the pyridinium-epichlorohydrin-polyethyleneimine condensation product of lignin). Lignin-containing-exchangers have a pronounced tendency to adsorb high-polymeric substances such as ligninsulphonic acid. The most effective adsorbent for the latter is the lignin condensation product containing 90% of alkali lignin and 10% polyethyleneimine; this absorbs 100.5% of its dry weight of ligninsulphonic acid. In general the nitrogen-poor products are better than the nitrogen-rich ones. The possible use of this action in purifying sulphite liquors from lignin leaving the sugars in solution is considered. The ability to adsorb ligninsulphonic acid increases greatly with the maximum swelling expressed in grams of water adsorbed per 100 grams of dry exchanger. The lignin-free polyethyleneimine condensation product adsorbs the most highly sulphonated ligninsulphonic acids; as the lignin content increases, the sulphur content of the adsorbed ligninsulphonic acid decreases. The adsorbed acid can be eluted with concentrated ammonia or 5% sodium hydroxide solution. Preparations with low swelling ability have negligible ability to adsorb ligninsulphonic acid, but the adsorbing ability for low molecular weight inorganic or organic acids is not affected. The anion-adsorbing capacity of the exchangers is essentially independent of the valence of the ion; all are adsorbed as univalent ions, but mutivalent ions are adsorbed preferentially to a slight but recognizable degree.

N. V. deBataafsche Petroleum Maatschappij, Dutch pat. 58,508 (Nov. 15, 1946); *C. A.* 41: 4597 (1947).

Finely divided straw, peat, sawdust, lignite, coal, dried sulphite waste liquor, and bagasse can be converted to ion exchangers by carbonizing with sulphuric acid.

12. AMMONIA-BASE SULPHITE PROCESS

Thode, E. F., and Lee, Y. H., *Tappi* 33: 257–260 (1950); C. A. 44: 8646 (1950).

A pre-impregnation cycle, as suggested by Richter, was employed in the sulphite cooking of mixed birch, beech, and maple chips. NH_3 base was used because of higher mobility and solubility than Ca^{++}. A typical cook involved steaming of chips for 1 hour at 15 p.s.i.; impregnation with 12% $(NH_4)_2SO_3$ for 2 hours at 240 to 270° F. followed by 5.5 to 7.5 hr. ammonia base sulphite cook at 300 to 320° F. and 100 p.s.i. Yields obtained were 44 to 49%; α-cellulose on wood basis, 37.5 to 39.4%. This process enables relatively rapid cooking because of elimination of extremely long impregnation time for Ca-base liquor on hardwood. Some data on reuse of the $(NH_4)_2SO_3$ liquor were obtained.

13. MAGNESIUM-BASE SULPHITE PROCESS

Hatch, R. S., U. S. pat. 2,308,364 (Jan. 12, 1943); *C. A.* 37: 3606 (1943).

In a cyclic process involving the cooking of wood with a relatively pure magnesium bisulphite cooking acid, removing and extracting the waste liquor from the cooking operation, concentrating and burning the waste liquor to recover heat from the organic material, magnesium oxide as an ash, and sulphur dioxide in the combustion gases, recombining the magnesium oxide ash and the sulphur dioxide in the combustion gases to form fresh cooking acid; soluble potassium salts derived from the wood are periodically removed from the ash by leaching it with water, and potassium and magnesium compounds are recovered from the resulting liquor.

Palmrose, G. V., and MacBain, D. K., U. S. pat. 2,320,294 (May 25, 1943); *C. A.* 37: 6459 (1943).

An arrangement of apparatus is described, and a cyclic method is employed for treating lignocellulose material which involves cooking the material under pressure with a solution of magnesium bisulphite containing a substantial excess of free sulphur dioxide, releasing gases during and after the cooking, separating the acid waste liquor from a treated cellulose material after completion of the cooking operation, evaporating and burning the separated acid waste liquor to produce an ash consisting mainly of magnesium oxide and combustion gases containing sulphur dioxide, adding magnesium compounds and sulphur as required to make up losses of the evaporated liquor during the burning of the waste liquor, making a slurry of the magnesium oxide ash, absorbing the sulphur dioxide in the combustion gases in the slurry to reform magnesium bisulphite to be used in a subsequent cooking operation, condensing the vapor resulting from the evaporation of the waste liquor to separate sulphur dioxide from it, absorbing the separated sulphur dioxide in the magnesium bisulphite liquor produced from the ash and the sulphur dioxide of the combustion gases, and returning the reformed magnesium bisulphite containing the absorbed sulphur dioxide for further cooking of lignin-cellulosic material.

Hatch, R. S., *Pacific Pulp Paper Ind.* 18: No. 1: 11–13 (1944); *C. A.* 38: 2203 (1944).

The status of sulphite waste liquor utilization is briefly outlined with special reference to the process of Tomlinson (*C. A.* 35: 4926; 36: 7246).

Wilcoxon, L. S., Can. pat. 420,622; *C. A.* 38: 3882 (1944); U. S. pat. 2,354,175 (July 18, 1944); *C. A.* 38: 6561 (1944).

An apparatus is described for recovery of heat and chemicals from waste liquors such as those from cellulosic pulp production with magnesium bisulphite.

Tomlinson, G. H., *Pulp Paper Mag. Can.* 45: 817–820 (1944); *C. A.* 39: 187 (1945).

The answer to the problem of sulphite waste liquor must meet the following conditions: (1) It must be one capable of being universally employed. (2) It should provide for the complete utilization of all the components in the liquor, both organic and inorganic. (3) It must present economic advantages. It is considered that all these requirements are fulfilled by the magnesium bisulphite process (*C. A.* 34: 4263).

Wilcoxson, L. S., *Paper Mill News* 68, No. 24: 104, 106, 108 (1945); *C. A.* 39: 5480 (1945).

A brief description of the magnesium bisulphite system (*C. A.* 34: 1484, 4263) as applied to an existing 100-ton per day sulphite mill, with a diagram of the layout of the pilot plant at the Cornwall, Ont., mill of Howard Smith Paper Mills, and a flow diagram of a 100-ton mill.

Tomlinson, G. H., U. S. pat. 2,385,955 (Oct. 2, 1945); *C. A.* 40: 207 (1946).

A detailed description of the recovery system of the magnesium bisulphite pulping process. The black liquor is neutralized with recovered magnesium oxide, concentrated to 45 to 70% solids and sprayed into a high temperature (2100 to 2400°F. or 1050 to 1215°C.) furnace, where the sulphur dioxide is released and recovered and the liquor solids are burned to a dry unsintered residue containing a relatively high proportion of caustic magnesium oxide. The dried residue is removed from the furnace continuously by flotation in the gaseous product of combustion at such a rate that the period spent within the furnace is insufficient to "dead-burn" the magnesium oxide. A portion of the dried residue is used to neutralize the fresh black liquor before concentration and the remainder is suspended in water which is then sulphited with sulphur dioxide, thus forming fresh pulping liquor (cf. *C. A.* 35: 4926).

Tomlinson, G. H., U. S. pat. 2,377,282 (May 29, 1945); cf. U. S. pat. 2,179,456 (*C. A.* 34: 1484); *C. A.* 40: 1034 (1946).

An apparatus is described for use in connection with the manufacture of sulphite pulp from cellulosic material with a pure magnesium-base cooking liquor. It aims at an efficient recovery of heat and chemical values from the residual pulp liquor.

Solvay Process Co., *Northeastern Wood Utilization Council Bull.* No. 7: 65–72 (1945); *C. A.* 40: 1655 (1946).

Calcium, magnesium, sodium, and ammonium bisulphite pulping are discussed. The specific advantages of the ammonium bisulphite process are pointed out.

Aries, R. S., *Northeastern Wood Utilization Council Bull.* No. 7: 65–72 (1945); *C. A.* 40: 2000 (1946).
A comparative study was made of the various methods of sulphite pulping from the point of view of acid manufacture, process efficiencies, and recovery methods applicable to the various bisulphite bases. A calcium bisulphite base deposits calcium sulphite in equipment as scale and in the chips, hindering penetration, resulting in non-uniform pulp, decreased yield, and scaling in waste liquor evaporators. Concentration is costly. The magnesium bisulphite base produces a pulp superior to calcium but inferior to sodium and ammonium bisulphite bases. Capital cost of recovery plant is high and some scaling occurs. Sodium bisulphite base produces the best pulp in good yield, provides exact control, eliminates scaling. Its waste liquor is readily combustible. Ammonium base uses ammonia gas and produces pulp superior to calcium and magnesium base. It gives a higher yield of uniformly cooked pulp, eliminates scaling, and waste liquors can be recovered as gaseous ammonia and sulphur dioxide and lignin useful in fertilizers. Consumption of ammonia is 92 pounds per pulp ton, of which 50% is recoverable. The flow sheet of the ammonium bisulphite process and a material balance of the cooking acid, digester, and recovery furnace are described.

Hatch, R. S., *Paper Trade J.* 122, No. 11: 54–56 (1946); *C. A.* 40: 2624 (1946).
A description is given of possible methods for disposing of or utilizing the organic solids in sulphite waste liquor. The cyclic process which depends upon change of base from calcium to magnesium with the evaporation and burning of the magnesium base waste liquor, and the recovery of heat and cooking chemicals is outlined. Modifications in carrying out the cooking process leading to greater steam economy, chemical engineering problems involved, and expected savings in sulphur and base are described.

Hatch, R. S., *Pulp Paper Mag. Can.* 47, No. 9: 80–84 (1946); *C. A.* 40: 6812 (1946).
Calcium, sodium, ammonium, and magnesium cooking liquors for the sulphite process, and methods for the recovery of the waste liquors are compared. Magnesium-base liquors offer the simplest and most efficient recovery possibilities.

Hatch, R. S., *Pulp Paper Mag. Can.* 48, No. 13: 85–87 (1947); *C. A.* 42: 1053 (1948).
A discussion of the recovery of chemicals and energy in the sulphite process through the use of magnesium as a base.

Pulliainen, E., *Finnish Paper Timber J.* 29: 225–229 (1947); *C. A.* 42: 3563 (1948).
The use of various bisulphite salts in the sulphite process is reviewed; the magnesium salt is the most satisfactory from a chemical and economic standpoint.

Port Ensui Industrial Co., Japan pat. 163, 146 (March 23, 1944); *C.A.* 42: 3962 (1948).
By adding 1.5% oxalic acid to the waste liquor of bagasse pulp prepared by the magnesium sulphite method and then heating, sulphur dioxide is expelled and magnesium, calcium, aluminum, and iron precipitated as oxalates.

Arnst, A., *Am. Forests* 55, No. 1: 16–17; *Chemurgic Digest* 8: 4–7; *Western Pulp and Paper* 2, No. 1; 6–9 (1949); C.A. 43, 2771 (1949).
A discussion of the magnesium-base pulping process.

Schwabe, K., Fiedler, H., and Schlosser, H., *Holzforschung* 3: 5–23 (1948); *C.A.* 43: 3196 (1949).
The pyrolysis of dried powdered spruce sulphite waste liquor (from calcium bisulphite digestion) in the presence of carbon dioxide at about 300°C. led to the recovery of a maximum of 72% of the sulphur as sulphur dioxide and hydrogen sulphide. Higher or lower temperatures resulted in smaller yields. Similar results were obtained in the presence of hydrogen (at 300°C.). Pyrolysis of a dried magnesium bisulphite waste liquor gave a recovery of over 80% sulphur at 400°C. and well over 90% when the decomposition was carried out at 800°. The residual magnesium oxide represents more than 80% of that used in the original cooking liquor. Magnesium sulphate, when heated with 16.5% magnesium base sulphite liquor for 1 hour at 625°, gave rise to 79.2 to 81.7% of the sulphur originally present. When it is pyrolyzed in the presence of 10.4% charcoal, however, only about 55% of the original sulphur was recovered. This represents the possibility of obtaining magnesium oxide and sulphur dioxide (for the magnesium bisulphite cooking liquor) from the relatively inexpensive magnesium sulphate.

Baker, R. E., and Hutton, F., *Pulp Paper Mag. Can.* 51, No. 6: 82–84 (1950); *C.A.* 44: 8646 (1950).
Initial operating experiences with the magnesium base sulphite process at Longview are reviewed. Make-up chemicals amounted to 100 pounds S and 62 pounds MgO/air-dry ton, with recoveries of 65% S and 75% MgO. Sources of chemicals and organic matter losses were analyzed. Heat recovery amounted to 8030 pounds steam at 700°F. and 615 p.s.i. plus 850,000 B.t.u./ton of pulp in the cooling towers.

14. LABORATORY INVESTIGATIONS

Erdtman, E., Ericson, P., and Hägglund, E., *Svensk Papperstidn.* 46: 121–127 (1943); *C. A.* 37: 4568 (1943).
An attempt was made to establish whether or not the lignin in wood is chemically combined with carbohydrates. Two sodium bisulphite cooks were carried out on crushed spruce wood chips and on shavings respectively. In the first the sulphite liquor was precipitated with *bis* (*p*-dimethylaminophenyl) methane and the precipitate after decomposition gave a ligninsulphonic acid which was similar in composition to the same fraction obtained from a technical sulphite waste liquor. It had a very low copper number before and after hydrolysis. The low molecular ligninsulphonic acids which were not precipitated had a copper number of 5.3 before and 67.5 after hydrolysis. This is explained by the fact that the relatively high pH of the cooking liquor prevents a more drastic hydrolysis. The increase of the copper number is not necessarily the result of the hydrolysis of carbohydrate-*bis* (*p*-dimethylaminophenyl) methane compounds, but may also come from sulphonated carbohydrates containing glycosidic linkages. The presence of the latter is proved by recent unpublished investigations. Since a large proportion of the reducing sugar is precipitated by basic lead acetate, they cannot consist of simple sugars. The waste liquor from the second cook was separated by *bis* (*p*-dimethylaminophenyl) methane into a high molecular ligninsulphonic acid which, apart from a higher sulphur content, had the same composition as that from the first cook. From the mother liquor two ligninsulphonic acid fractions were obtained by fractional precipitation with brucine. From the mother liquor of the brucine precipitates, after treatment with barium hydroxide and passage through an organolite charged with hydrogen ions, the recovered barium salts of the low molecular ligninsulphonic acids were precipitated with alcohol and had a copper number of 9.1 before and 72 after hydrolysis. Only small amounts of mannose (about 6%) were isolated from the last mother liquors. The ligninsulphonic acids of the brucine precipitations are probably contaminated with carbohydrate sulphonic acids or combined with such acids by glycosidic linkages.

Schenck, W. A., *Paper Trade J.* 117, No. 14: 97–100 (1943); *C. A.* 37: 6890 (1943).
A critical discussion is given of the sulphonation of lignin in the sulphite cooking process on the basis of Freudenberg's structural formula for lignin. Ligninsulphonic acids from two widely different cooks were fractionated by chemical and physical methods and the fractions were further investigated to determine whether lignin is sulphonated by entrance of several sulphonic acid groups into the molecule without degradation of the latter, or whether the lignin molecule is broken into smaller fragments each having one sulphonic acid group. The results so far obtained indicate that a breakdown of the lignin molecule into smaller sulphonated fragments takes place.

Schwabe, K., and Preu, E., *Cellulosechem.* 21: 1–6 (1943); *C. A.* 38: 2202 (1944).
The thiocyanogenation of ligninsulphonic acid was studied. Ammonium ligninsulphonate was prepared from a fermented spruce sulphite waste liquor by precipitation of the calcium with ammonium carbonate. Since thiocyanogen is not very stable and in a nascent state apparently reacts by substitution, the thiocyanogenation was carried out by passing Br or Cl into a solution of sulphite waste liquor and ammonium thiocyanate at 0 or 15°. The thiocyanogenated ammonium ligninsulphonate was isolated by precipitation with an alcohol, butyl giving a higher yield than ethyl or methyl. Instead of liberating thiocyanogen by means of halogen, the thiocyanogenation may also be effected electrolytically. From the analyses it is calculated that one hydrogen of each benzene ring in the lignin molecule is substituted by a thiocyanogen group.

Pearl, I. A., *Pacific Pulp Paper Ind.* 17, No. 9: 32–34, 36, 39 (1943); *C. A.* 38: 2203 (1944).
A study was made of the effects of concentrations varying from 1:400 to 1:100,000 of sulphite waste liquor in sea water, both with and without aeration. Concentrations of less than 1:5000 have no effect on the chemical constituents of the sea water.

Schröter, G. A., *Papier-Fabr. Wochbl. Papierfabr.* 1943, No. 2: 50–54; *C. A.* 38: 2486 (1944).
The application of ultrachromatographic adsorption to spruce and beechwood sulphite waste liquors is discussed, together with the correlation between ultraviolet fluorescence and ligninsulphonate content of such liquors. The adsorbents used were aluminum oxide (prepared according to Brockmann and buffered by moistening with citrate solutions), Floridin XXF and Frankonit Kl. Pure glucose and xylose were slightly absorbed

by Floridin from aqueous solution, but only glucose was sorbed to a slight extent from a spruce sulphite waste liquor diluted 1 to 10, the reducing value dropping from 0.23 to 0.21%. When filtered through Frankonit Kl, the dilute spruce sulphite waste liquor yielded a sulphur-containing fraction that showed a very weak blue fluorescence and which, although giving a positive trypaflavin reaction, gave no test with β-naphthylamine hydrochloride. This indicated the possible presence of a nonfluorescent β-ligninsulphonic acid. This fraction was precipitated with basic lead acetate, the precipitate washed to remove mannose (identified as the phenyl hydrazone), and decomposed with 5% sulphuric acid. The resulting lead sulphate was washed on the centrifuge and the combined liquors neutralized with barium hydroxide. Thus a barium salt was obtained which however was not homogeneous. When a beechwood sulphite liquor was passed through alumina, buffered to a pH of 5, several ultraviolet fluorescent zones were noted, one of which was intensely yellow. By extraction with 2% ammonium hydroxide, the substance responsible for this yellow fluorescence was finally isolated as a compound high in ash and also containing nitrogen and about 1.6% sulphur. When spruce sulphite waste liquor was passed through alumina, several strongly fluorescing zones formed. One of these initially showed an intense white-blue-violet fluorescence which disappeared on contact with sulphur dioxide but returned on rinsing with water. Similar reactions and sorption were noted with a calcium ligninsulphonate that had been precipitated by alcohol from spruce sulphite waste liquor. However these reactions are not proof that the original fluorescing material was actually a ligninsulphonate. Alumina adsorbs very little reducing sugar from spruce waste sulphite liquor, the latter behaving very similarly on fermentation, whether filtered through alumina or not.

Aulin-Erdtman, G., *Svensk Papperstidn.* 47: 91–99 (1944); *C. A.* 38: 3467 (1944).
The ultraviolet absorption spectra of a number of lignin preparations and lignin model substances have been determined. The results indicate that the position of the maximum (around 280 mmu) and the minimum (around 260 mmu) of barium α-ligninsulphonate, obtained from a rayon pulp cook, is approximately characteristic of a macromolecule built up from dihydroeugenol via dihydro-dehydro-diisoeugenol. The slow decrease of the absorption intensity of lignin derivatives toward longer wave lengths cannot be explained by a furan or pyran structure. On the contrary, it requires the presence of other systems of conjugated double bonds, at least in the isolated lignin. Other comparisons do not support Freudenberg's assumption that ligninsulphonic acids are formed by opening of coumaran rings. The absorption curves of barium salts of α- and β-ligninsulphonic acids from two extremely different waste liquors were of the same general type. The intensity of the absorption is proportional to the methoxyl content of the various preparations. It can be concluded therefore, that the β-acids which are low in methoxyl contain an absorbing constituent of normal lignin type together with a non-absorbing methoxyl-free substance. Whether these constituents are chemically combined or not cannot be decided by spectrochemical methods alone.

Aulin-Erdtman, G., *Svensk Kem. Tid.* 55: 116–122 (1943); *Papier-Fabr. Wochbl. Papierfabr.* Auszuge 1943, 229; *C. A.* 38: 4127 (1944).
On the basis of reactions of phenylpropyl derivatives, Freudenberg's formula for spruce lignin is discussed. Through comparison of the course of the bromination of lignin with that of cresol and dihydroeugenol, it is proved that the formation of 6-bromovanillin by bromination and oxidation of lignin does not indicate that phenolic hydroxyl groups in the lignin building stones are etherified. Alkylguaiacols condense in the 5-position as shown with eugenol alcohol and with some 2- and 3-substituted 5,5′-dialkyldiphenylmethanes. 2,2′,3,3′-tetramethoxy-5,5′-diallyldiphenylmethane, on oxidation with permanganate, gives up to 59% isohemipinic acid. From this result it would be expected that, on the basis of Freudenberg's lignin formula, considerably higher yields of isohemipinic acid should be obtained on oxidation of methylated ligninsulphonic acids.

Doering, H., *Papier-Fabr. Wochl. Papierfabr.* 1943, 215–217; *C. A.* 38: 4128 (1944).
A new very rapid method for the determination of acetic and formic acids in beechwood sulphite waste liquor which depends upon the extraction of a 10-ml. sample (acidified) with a large volume of ether and titration of the extract with 0.1 *N* sodium hydroxide. The results, calculated to acetic acid, agree well with those obtained by distillation.

Noll, A., *Papier-Fabr. Wochbl. Papierfabr.* 1944, 39–42; cf. *C. A.* 32: 2742; *C. A.* 39: 1756 (1945).
New methods for the detection of ligninsulphonic acid depend upon the use of "Surfen" (bis(2-methyl-4-amino-6-quinolyl) carbamide dihydrochloride) which is not a dyestuff but which gives a voluminous cream colored precipitate with ligninsulphonic acid. Examined in the light of a special lamp (described) the limit of sensitivity of this test is 1.9 ligninsulphonic acid per 0.045 ml. Other excellent but less sensitive reagents which give precipitates are the purified basic dyes "Astraphloxin FF" (red ppt.) "Astraviolet FF" (violet ppt.), tryptoflavin (yellow ppt.), safranins T and MN, phenosafranine and methylene violet 3RA (red ppts.). Other basic dyes were also tested of which benzoflavin was the least sensitive. "Surfen" is suggested as a reagent for the determination of ligninsulphonic acid.

Schwabe, K., *Papier-Fabr. Wochbl. Papierfabr.* 1944, 115–123; *C. A.* 39: 1758 (1945).
The liquors examined by physicochemical means included fermented spruce sulphite waste liquor, and unfermented (purified and untreated) beechwood sulphite waste liquors. Dried powders prepared from these solutions were also studied. The color changes undergone on concentrating sulphite waste liquors were followed by the Lange photoelectric colorimeter and absorption was plotted against liquor concentration. For a specific liquor, light absorption is a function of the concentration, even when the concentrations are fairly high. Different liquors give rise to different absorption curves. With rising pH (between 3 and 9) absorption increases greatly. The pH has a greater influence at lower than at the higher concentrations. Heating of either the solutions or the dry powders causes a marked increase in absorption. The longer the heating period, and the higher the temperature, the greater is this effect. The viscosity of the liquors increases with falling temperature. The viscosity of the liquor from spruce was higher than that of the beech liquors and these differences are discussed. At higher temperatures, the viscosities are not seriously affected by pH. The liquors were evaporated and the residues were then heated at various temperatures. When the heated solids were redissolved to form 40% solutions the latter showed viscosity differences. The greater the preheating temperature, the greater was the rise in viscosity. The "molecular weights" of the solids from spruce liquors were considerably higher than those from beechwood liquors. When powdered ligninsulphonic acids were heated and then dialyzed, the molecular weights of the solids increased with the temperature and with the time of heating as well as with the length of the dialysis period. The molecular weight increases were greater with spruce than with beechwood ligninsulphonates.

Erdtman, H., *Svensk Papperstidn.* 48: 75–81 (1945); *C. A.* 39: 2196 (1945).
Sprucewood (extracted) was sulphonated with a mixture of sodium sulphite and sodium bisulphite to yield 84% woody residue containing 64% of the methoxyl of the original wood. After acidification with dilute hydrochloric acid, and washing until the filtrate was free of chlorine ions, the woody residue was extracted three times with water at 80 to 90°C. to give three filtrates A, B, and C. Each was neutralized with barium hydroxide, centrifuged, concentrated, and the barium salts precipitated by pouring into ethanol. The salts were then converted into the free acids by means of an organolith, and neutralized with a hot concentrated alcoholic solution of bis-dimethylamino-diphenylmethane giving the salts of this base which were filtered and decomposed with dilute sodium hydroxide. After extraction with ether, the sodium ligninsulphonates were again converted into the free acids. The ligninsulphonic acid corresponding to extract A was repurified by precipitation with bis-dimethyl-aminodiphenylmethane followed by precipitation as the barium salt. The latter had 9.4% barium, 4.1% sulphur, 13.8% methoxyl, a ratio of S to MeO of 1:3.5, and a copper number of 1.8 before and 7.3 after hydrolysis. It thus contains 4 S per 7 C_{20} atoms or 3 "unchanged" C_{20} units which are not sulphonated. The acid was converted to the calcium salt and resulphonated with calcium bisulphite. The barium salt of the ligninsulphonic acid so formed had Ba 15.2, S 6.1, MeO 12.2%, a S to MeO ratio of 1:2.1, and a copper number of 3.3 before hydrolysis and 6.8 after. The ultraviolet absorption spectra of the ligninsulphonic acids before and after this second sulphonation were determined and compared with that of isolated native lignin from black spruce. The curves show that no fundamental change in the lignin molecule has taken place. Further sulphonation of the lignin probably does not involve such deep-seated changes of the structure of the lignin molecule as the scission of coumarane rings. The fact that under ordinary conditions lignin cannot be sulphonated beyond 1 S per 2 MeO groups may indicate that lignin is built up to C_6—C—C—C—C—C_6 units. The formation of vanillin

may be explained by oxidative scission of the side chain having an oxygen atom at the carbon atom neighboring the benzene ring.

Lautsch, W., and Piazolo, G., *Cellulosechem.* 22: 48–54 (1944); cf. *C. A.* 38: 6091; *C. A.* 39: 2403 (1945).
The ligninsulphonic acid fractions from two different sulphite cooks were studied, one from a soft commercial cook and the other from a medium-hard laboratory cook. In either case the lignin components were precipitated in nine successive fractions by means of benzacridine. In both series the first precipitate yielded fractions lower in sulphur and the final precipitates fractions higher in sulphur. Extensive analytical data for C, H, N, and MeO and S are given. The first precipitates contained approximately 1 S per 20 C atoms; the final, 1 S per 10 C atoms. The ratio of S to MeO was 1:2 in the first and 1:1 in the final fractions. Evidently a uniform distribution exists between methoxyl and carbon in spruce lignin. In order to dissolve lignin from spruce wood at least one sulphonic acid group per 20 carbon atoms is required. Inasmuch as individual ligninsulphonic acid fractions (when computed to lignin) showed no significant differences attempts to separate α- and β-lignin were omitted. The sulphite waste liquors contained no lignin-carbohydrate complexes. Analyses involving methoxyl balances showed that about 10% of the methoxyl originally present in the wood is not a component of lignin. Lignin fractions when oxidized with nitrobenzene gave varying vanillin yields. The fractions lowest in sulphur gave the lowest yields (13.8–16.5% calculated to sulphur-free lignin) and those highest in sulphur gave the highest vanillin yields (26.7%). The low average vanillin yield of 17% is due to the fact that the ligninsulphonates low in sulphur predominate in amount.

Höpner, T., *Cellulosechem.* 22: 33–48 (1944); *C. A.* 39: 2403 (1945).
By withdrawing liquors at hourly intervals during an indirect rayon sulphite cook, the changes in liquor composition and in the stock were followed. The maximum fermentable sugar content was 2.3% and the maximum mannose content 1.6% after 13 hours, both of which remained substantially the same thereafter. Galactose reached its maximum (0.4%) after 11 hours and then fell off quite rapidly. The maximum pentosan value (1.1%) was reached after 10 hours and then dropped slowly. Glucose values are questionable but reached about 0.6% at 13 hours. Besides the simple sugars, the liquors (removed after 8 to 9 hours' digestion) contained polysaccharides which were purified as follows: Calcium was removed either as $CaSO_4$ or $CaCl_2$ (the latter being soluble in aqueous alcohol). The polysaccharides were precipitated with alcohol, redissolved in water, and freed from lignin derivatives with trypaflavin and the filtrate treated with ammonium oxalate and ammonium hydroxide to precipitate residual trypaflavin and calcium. The aqueous filtrate was acidified, precipitated with alcohol and repeatedly dissolved in water and reprecipitated with alcohol. The resulting polysaccharide was an amorphous powder with a degree to polymerization of 127 (osmotic pressure) and was substantially free of lignin. On hydrolysis, sugars were obtained of which only about 1% was pentose, about one third was mannose and the remainder probably glucose. Precipitation of the sulphite waste liquor with trypaflavin gave a lignin sulphonic acid complex that was practically sugar-free and evidently the sulphite cook destroys lignin-hemicellulose compounds. Analyses show that in the lignin-trypaflavin precipitate there are three N atoms for every S atom, i.e., every sulphonic acid group binds one molecule of trypaflavin when analyses of the precipitate are calculated back to lignin. The values check with those found by Freudenberg for Cuproxam lignin.

Freudenberg, K., Lautsch, W., and Piazolo, G., *Cellulosechem.* 22: 97–102 (1944); *C. A.* 40: 3258 (1946).
The action of sodium bisulphite cooking liquor on spruce wood at 70° was studied. The digestion process was repeated eight times, and eight waste liquor fractions thus obtained. Each fraction was purified by passing through a cation exchanger and precipitating with benzacridine-HCl. The resulting precipitate was decomposed with alkali, the base extracted with ether, and the ligninsulphonic acid liberated by passing through a cation exchanger. From carbon, hydrogen, sulphur, and methoxyl analyses, it was concluded that a part of the lignin requires one sulphonic acid group per 2–3 lignin building stones, another part required 3–5 sulphonic acid groups per 4 lignin building stones in order to render the lignin soluble. The carbon to methoxyl ratio of all fractions is 10–12:1. The hydroxyl content of 8.6% in the ligninsulphonic acid from the first fraction decreases with increasing sulphur content to 5.03% in the ligninsulphonic acid of the last fraction. The phenolic hydroxyl content decreases from 1.8% in fraction 1 to 1.4% in fraction 6. This fact indicates that the phenolic hydroxyl content is not affected by the degree of sulphonation. Potentiometric titration and equivalent weight calculations from the ratio of sodium to sulphur in the sodium ligninsulphonates indicate the presence of one carboxyl group for equivalent weights of 1500 to 3000. The analyses on a sulphonic acid-free basis agree very well with those found for cuprammonium lignin. Fractions 5–8 show a considerable decrease in the ether oxygen as compared with that of fractions 1–4. These results and the fact that with increasing sulphonation the phenolic hydroxyl content does not increase, show that in the sulphonation of lignin at 70°, no benzofuran rings are split but that ether linkages, as they occur in pinoresinol, are split without formation of new phenolic hydroxyl groups.

Shinn, D. L., *Pulp and Paper Ind.* 19, No. 12: 66–68 (1945); *C. A.* 40: 4300 (1946).
Sulphite waste liquor was electrolyzed with platinum electrodes at 20 and 80° with 0.2 ampere current. At 20°, the loosely combined sulphur dioxide approached a constant value of 1.3 grams per liter (7-day experiment), whereas at 80° it constantly decreased (from 5.08 to 0.27 grams per liter in 6 days). The lignin content (as determined by precipitation with 2-naphthylamine) appeared to increase, possibly through a polymerization of a portion of the lignin. The stability of the sulphonic acid group is shown by the fact that less than 7% is decomposed at 80° in 6 days. The quantity of carbon dioxide evolved represented 39% of the theoretical (based on loss of solids). Details are given of the electrolysis (63 days) of a liquor which had been neutralized and passed through a cation-exchange resin; the reducing sugars decreased from 31.6 to 20.1 grams per liter; the percentage decrease in total solids and ligninsulphonic acid was about the same. The increased quantities of formic and acetic acids, methanol, and ethanol probably represent intermediate products in the decomposition of complex organic compounds in the liquor. Separation of the anode and cathode chambers by a porous diaphragm did not produce any appreciable separation of liquor constituents through differences in ion velocities. No significant reaction took place at the cathode during the electrolysis. During the 63-day experiment, the methoxyl content of the lignin decreased from 14 to 1.8%.

Pennington, D., and Ritter, D. M., *J. Am. Chem. Soc.* 68: 1391–1392 (1946); *C. A.* 40: 5245 (1946).
A study was made of the oxidation of ligninsulphonic acids by periodic acid. The following methods were used for purification of the ligninsulphonic acid prior to oxidation: (a) fermentation with yeast; (b) diffusion of fermented sulphite waste liquor in sintered-glass diffusion cells for 62 days; (c) preparation of the barium salt of ligninsulphonic acid, soluble in 40% aqueous acetone, insoluble in 70% aqueous acetone; (d) dialysis of fermented sulphite waste liquor against running water for 168 hr.; (e) purification of sulphite liquor through the quinoline salt. The methoxyl content of the ammonium salts and the equivalent weight per mole of periodate reduced were (a) 8.5%, 200; (b) 10.1%, 304; (c) 9.7%, 347; (d) 13%, 525; (e) 11.9%, 525. The first two are believed to be substantially free of carbohydrate material The equivalent weight of 525 corresponds approximately to a ratio of two methoxyl groups for each linkage oxidizable by periodic acid.

Erdtman, H., Aulin-Erdtman, G., and Lindgren, B., *Svensk Papperstidn.* 49: 199–200 (1946); *C. A.* 40: 6812 (1946).
Ligninsulphonic acid with a low degree of sulphonation (1 S: 40 C) was prepared by a prolonged cook of spruce wood meal at 135° with sodium sulphite at pH 5. The isolated sulphonic acid was found by acetylation to contain two hydroxyl groups per C_{10} unit, which is more than previously assumed. On further sulphonation, hydroxyl groups disappear, evidently through esterification or through a reaction similar to that for hydroxy ketones and sulphite. Ligninsulphonic acid obtained by the above conditions and also from sulphite waste liquor resulting from the production of a strong pulp, could be further sulphonated toward a value of 1 S:10 C. No further sulphonation could be obtained of ligninsulphonic acid isolated from sulphite waste liquor resulting from rayon-pulp cooks. Thus, the latter cooking conditions, characterized by high acidity and temperature, affect the further reactivity of lignin. The ultraviolet absorption of the different ligninsulphonic acids is not materially changed, even when the sulphonation is carried on until 1 S:10 C is obtained.

Adler, E., *Svensk Papperstidn.* 49: 339–346 (1946); *C. A.* 40: 7621 (1946).
An appreciable part of the simple sugars formed in sulphite pulping by hydrolysis of the hemicelluloses is rendered nonfermentable through irreversible conversion to aldonic acids under the influence of the cooking acid (*C. A.* 24: 4268). In addition part of the sugars are changed into sugar sulphonic acids which are stable in acid and alkaline solution and cannot be reconverted to the original sugar. Both aldonic acids and sugar sulphonic acids are present in sulphite waste liquor but they are difficult to separate from ligninsulphonic acids. Holocellulose was pressure heated with sulphite cooking acid and the sulphonic acids thus obtained contained only a small amount of ligninsulphonic acids (*C. A.* 38: 5082). The latter were almost completely eliminated by passing the free acids through an an-

ion exchanger. Such material does not absorb ligninsulphonic acids of high molecular weight and the sugar acids are eluted with barium hydroxide solution. Analyses show these barium salts to be a mixture of aldonic acids and sugar sulphonic acids. Their separation into individually pure compounds is difficult and is presently studied by using special techniques with anion exchangers. Glucose, when subjected to sulphite cooking conditions, was converted to an aldonic acid but no sulphonic acids were found. However, when glucose was heated with sodium bisulphite-monosulphite at pH 6, two sulphonic acids were formed which were separated by formation of their phenyl hydrazone barium salts. From a study of their reactions and behavior on oxidation, these acids were found to have the formulas $C_6H_9O_8SO_3H$ and $C_6H_{11}O_5SO_3H$ respectively. Since the ratio of carbon to oxygen is the same as for glucose, these carboxylic acids must have been formed by a mechanism of intramolecular exchange of oxygen. This indicates similarity to saccharinic acids and a tentative building scheme of similar type is presented for such sulphosaccharinic acids to explain the groupings found in the carbohydrate molecules. It is suggested that further study of such sulphonic acids may also contribute to a better understanding of the sulphonation mechanism of lignin.

Zeidenberg, B. S., *Legkaya Prom.* 1946: No. 7/8: 35; *C. A.* 41: 2241 (1947).
A colorimetric method for the determination of sulphite waste liquor based on the blue color produced in alkaline solution with phosphotungstomolybdate reagent. The content of sulphite waste liquor that can be determined ranges from 0.0001 to 0.025%. The optimum concentration for determination is 0.001 to 0.01%.

Eng, H., Österlof, J., and Sillén, L. G., *Svensk Papperstidn.* 49: 497–498 (1946); *C. A.* 41: 3293 (1947).
A method is described for the determination of sulphate in sulphite waste liquor which prevents oxidation of sulphur dioxide compounds. Formaldehyde and acetic acid are added to the sample prior to precipitation with barium chloride and the resulting precipitate purified in one of several ways described.

Samuelson, O., and Öhgren, T., *Svensk Papperstidn.* 49: 499–502 (1946); *C. A.* 41: 3294 (1947).
A simplified modification of the method worked out by Eng, Österlof, and Sillén for determining sulphate in sulphite waste liquor is described. Various modifications were investigated showing by experimental data that the above procedure is sufficiently accurate and reproducible also between different analysts. Results with the method used by different analysts on different types of sulphite waste liquor are given.

Samuelson, O., *Svensk Papperstidn.* 49: 575–581 (1946); *C. A.* 41: 3294 (1947).
The sulphate content of sulphite cooking liquor has been determined in samples taken from digesters during various stages of the cook and during the displacement, in the digester, of strong sulphite waste liquor with either dilute sulphite waste liquor or water. The results indicate a high dissolved sulphate content in the first part of the cook and that the sulphate content decreases before the maximum temperature is reached. This is taken to mean that calcium sulphate, the solubility of which decreases as temperature increases, actually separates during the cook. Some preliminary tests have shown that sulphite waste liquor can keep in solution, either as supersaturation or through specific solubilizing action of organic matter, more than ten times as much calcium sulphate hydrate as can pure water. The practical significance of the findings is discussed. For subsequent efficient evaporation of sulphite waste liquor, it is important to obtain liquor in which the ratio of sulphate to dry solids is as low as possible. Formation of sulphate during the cooking process to such extent that actually some sulphate separates shows that efforts to reduce sulphate content in the fresh cooking liquor are useless. Displacement washing in the digester should be carried out at the highest practical temperature, since this will reduce the amount of sulphate redissolved in the liquor which is to be evaporated.

Peniston, Q. P., Felicetta, V. F., and McCarthy, J. L., *Anal. Chem.* 19: 332–334 (1947); *C. A.* 41: 4308 (1947).
A method for the determination of sulphate in sulphite waste liquor by a conductometric titration is described.

Pennington, D., and Ritter, D. M., *J. Am. Chem. Soc.* 69: 665–668 (1947); *C. A.* 41: 5001 (1947).
Free diffusion over successive intervals was used to obtain a sequence of average intergral diffusion coefficients descriptive of the heterodisperse and polymeric nature of the ligninsulphonic acids. Diffusion coefficients found are: those based on total solids, diffusing 0.26–0.17, of methoxyl bearing components 0.16–0.12, of sulphur-bearing components 0.32–0.18, of periodate-oxidizable components 0.36–0.24 cu. cm. per day. Molecular weights were approximated by application of the Sutherland-Einstein equation. The values obtained indicate 60% of the ligneous material to have molecular weights of 10,000 or over with a minor fraction as low as 3000.

Zeidenberg, B. S., *Zavodskaya Lab.* 12: 914–918 (1946); *C. A.* 41: 5716 (1947).
A method is described for the determination of sulphite waste liquor in electrolytic copper baths The method is based on the fact that the phosphotungstomolybdate reagent gives a intensely blue color with lignin in alkaline solution and is suitable for sulphite liquor contents of 0.001 to 0.05%.

Lange, P. W., *Svensk Papperstidn.* 50, No. 11B: 130–134 (1947); *C. A.* 41: 7112 (1947).
A preliminary report is given of the course of removal of lignin in a sulphite cook, which was followed by taking samples after various periods of digestion. These samples were examined under the microscope and by ultraviolet absorption curves. The results show that the sulphonation of the lignin in the fibers occurs without the disappearance or the formation of an unsaturated system in conjugation with the benzene ring. It is very improbable that oxygen rings of the coumarane type are opened during the sulphonation. The distribution of the lignin in the fiber is discussed and it is found that the middle lamella is attacked by the cooking liquor at an early stage of the cook. The change of the dichroism of the lignin in the middle lamella during the cook was studied. From the results it seems that the lignin is not only the cementing material for the fibers but that this cementing effect is a function of the orientation of the lignin in the middle lamella.

Kratzl, K., and Bleckmann, C., *Monatsh.* 76: 185–199 (1947); cf. *C. A.* 40: 4042 (1946); *C. A.* 41: 7112 (1947).
A study was made of the bromination of ligninsulphonic acid and its model substances. Propioguaiacone and α-propioguaiacone were brominated in chloroform solution to yield 5,α-dibromopropioguaiacone. Sodium propioguaiacone α-sulphonate gave 5,α,α-tribromopropioguaiacone. Degradation of these two bromo compounds with oxygen (cobaltic hydroxide catalyst) gave 15 to 30% yields of 5-bromovanillic acid. Sodium α-propioveratrone sulphonate gave on bromination 6,α,α-tribromopropioveratrone which on degradation yielded 6-bromoveratric acid. Barium cinnamaldehyde hydrosulphonate and sodium phenyl-acetone-sulphonate did not yield nuclear bromine-substituted derivatives. Sodium ligninsulphonate (prepared from a commercial sulphite waste liquor through the quinoline salt) gave on bromination a chloroform-insoluble product and a chloroform-soluble material. The former contained about 4% bromine and on oxidation gave slight yields of phenols (mono- and dibromo-vanillins) and brown amorphous acids. The chloroform-soluble material contained no sulphur and could be separated into a neutral (largely bromoform), an acid, and a phenolic fraction.

Adler, E., *Svensk Papperstidn.* 50, No. 11: 261–266, No. 11B: 9–16 (1947); *C. A.* 41: 7113 (1947).
A study was made of the nature of the combination of loosely combined sulphurous acid in sulphite waste liquors and its behavior on chemical treatment. On neutralization of sulphite liquor to pH 6 with lime at 100°, only part of the loosely combined sulphurous aicd is split off, the remainder being removable only above pH 7. The sulphurous acid is apparently present as an aldehyde bisulphite addition compound. Sugar, furfural, glucuronic acid, and sugar sulphonic acid were found to combine only in insignificant quantities with sulphurous acid. Hydroxymethyl furfural could not be detected in sulphite waste liquor. Methylglyoxal, formaldehyde, and trioses add sulphurous acid to the extent of almost 100%. In rayon pulp cooks, the major part of the loosely combined sulphurous acid is combined with volatile aldehydes (e.g., formaldehyde and methylglyoxal) whereas in hard cooks a considerable part is attached to non-volatile carbonyl compounds. Sulphite waste liquor was freed of loosely combined sulphurous acid and treated with 2,4-dinitrophenylhydrazine. The corresponding hydrazone of ligninsulphonic acid was thereby obtained. It was found to contain one hydrazone grouping per eight methoxyl groups. No precipitate was obtained on similar treatment of fresh sulphite waste liquor. Thus, it was assumed that ligninsulphonic acid is present as a bisulphite addition compound in fresh sulphite liquor.

Yorston, F. H., *Pulp Paper Mag. Can.* 48: 13, 74–80 (1947); *C. A.* 42: 1054 (1948).
In a report on the analysis of sulphite waste liquor, complete details are given of recommended methods for total solids, sulphated ash, pH and buffer capacity, calcium, sulphur, methoxyl, strong acids, and a dye test for sulphonic acids (with azodimethylaniline). Additional methods include volatile organic acids, sulphur dioxide, sulphate, reducing and fermentable sugars, ethanol yield, fermentable sugars by sorption on yeast, and furfural yield.

Schwabe, K., and Hahn, E., *Holzforschung* I: 42–47, 79–89 (1947); *C. A.* 42: 1420 (1948).
Concentrated purified sulphite waste liquor (35% solids) was separated into 15 fractions by means of propanol. The lignin-sulphonic acids of the first 6 fractions had a molecular weight of 330; thereafter the molecular weight increased steadily to 26,200 (last fraction). A fermented sulphite liquor (50% solids) was similarly separated into 11 fractions, the molecular weights increasing from 360 to 16,900. The various fractions were tested for their reducing values, hygroscopicity and precipitability with Surfen No. 11, (*C. A.* 39: 1756) and analyzed for sulphur and methoxyl. The sulphur content decreased from 11% in the first fraction to 3.6% in the fraction with the highest molecular weight. However, the methoxyl content increased from 7.2 to 12.1%. The yields of the various fractions are given in tables and curves. Some of the fractions were converted into their barium and ammonium salts; the behavior with lead acetate is reported.

Ernsberger, F. M., and France, W. G., *J. Phys. and Colloid Chem.* 52: 267–276 (1948); *C. A.* 42: 2491 (1948).
Ligninsulphonic acid was prepared from the waste liquor of a sulphite pulp process by using ion exchange. By means of fractional diffusion, three fractions were obtained in order to examine the effect of molecular size on surface activity, degree of sulphonation, ratio of hydrogen ion to sulphur and other properties. Studies of the adsorption isotherms on portland cement, for instance, indicate a pronounced increase of surface activity with increasing molecular weight. The average molecular weights of two of the fractions were estimated from diffusion measurements to be 9500 and 2140. No constant value could be obtained for the lowest one, because of slow polymerization. The molecular weight varied between an initial value of 250 and 920 resulting 13 days after preparation. The second subject investigated was the dissociation of the acid and of its sodium, ammonium, barium, and calcium salts. Continued measurements led to moderate degrees of dissociation (20 to 60%) in contrast to the complete dissociation of sulphonates of low molecular weight.

Peniston, Q. P., and McCarthy, J. L., *J. Am. Chem. Soc.* 70: 1329–1332 (1948); *C. A.* 42: 4745 (1948).
Examination of a sample of purified ligninsulphonic acid by conductometric and potentiometric titration methods indicates the absence, or the presence in only low concentrations, of free phenolic hydroxyl groups in the material as it exists in sulphite waste liquor. By mild alkaline hydrolysis of ligninsulphonic acid, weakly acidic groupings are formed which show a pH value of about 9 and are regarded as phenolic hydroxyl groups. Periodate oxidation studies and the extent of nitrosolignin formation, as well as conductometric analysis, indicate phenolic hydroxyl liberation by mild alkaline treatment of ligninsulphonic acid; about 0.67 mole of phenolic hydroxyl per mole of methoxyl is liberated. The presumed remaining phenolic groups (0.33 mole per mole methoxyl) thus appear to be bound in a more stable type of linkage. Alkaline hydrolysis of ligninsulphonic acid brings about a decrease in molecular weight of some fractions of the acids. This and other evidence indicates that more than one type of bond serves to combine structural units in ligninsulphonic acids.

Samuelson, O., and Westlin, A., *Svensk Kem. Tid.* 59: 244–253 (1947); *C. A.* 42: 5220 (1948).
The loosely bound sulphur dioxide in sulphite waste liquor is the difference between the total titratable (with iodine) sulphur dioxide obtained on distillation and that determined by direct titration of the liquor. The loosely bound sulphur dioxide is defined as the value obtained by making the distillate alkaline and then rapidly acidifying the solution and titrating with iodine. The loosely bound sulphur dioxide is combined in the waste liquor with volatile aldehydes and other carbonyl and organic compounds as α-hydroxy sulphonic acids. The main volatile aldehydes are furfural, acetaldehyde, formaldehyde, and methylglyoxal. The possibility that ligninsulphonic acid in the waste liquor contains loosely bound sulphur dioxide is mentioned. On distilling the waste liquor, the volatile carbonyl compounds recombine with the distilled sulphur dioxide to form α-hydroxysulphonic acids in the distillate.

Olleman, E. D., Pennington, D. E., and Ritter, D. M., *J. Colloid Sci.* 3: 185–195; (1948); *C. A.* 42: 6206 (1948).
Crude ammonium ligninsulphonate was de-ashed with a cation exchange resin and converted to the barium salt. The barium ligninsulphonate was separated into three fractions by adding progressively increasing amounts of acetone to the aqueous solution. The middle and last fractions were de-ashed with a cation-exchange resin and further fractionated by a technique based on the formation of the quinolinium salt. This separated the sulphonates into three fractions preferentially soluble in water, quinoline, and ether respectively. The quinoline-soluble fractions, which represented the major portion, were subjected to diffusion analysis. Relative viscosities were also determined so that the shape factor could be calculated. The molecular shape was then used in calculating an approximate molecular weight from the diffusion data. The sulphonate obtained from the last fraction of the acetone fractionation was rather uniform in molecular weight with an average molecular weight of approximately 1500. The sulphonate obtained from the middle fractions contained material of molecular weights broadly distributed from 1500 up, with the average approximately 2,000.

Hachihama, Y., Shinra, K., and Kyogoku, Y., *J. Soc. Chem. Ind. Japan* 47: 209–215 (1944); *C. A.* 42: 7041 (1948).
Conductometric titrations were carried out on the digesting solution of sulphite pulp, its waste liquor, and the components of the liquor. The composition of the digesting liquor can be determined easily and quickly by conductometric titration. This permits the determination of the point of completion of digestion. When cooked at high pressure, sugars yielded organic acids of a high degree of dissociation. The titration curve of ligninsulphonic acid showed turning points due to the sulphonic acid group and phenolic hydroxyl group.

Stockman, L., and Hagglund, E., *Svensk Papperstidn.* 51: 269–274 (1948); *C. A.* 43: 395 (1949).
Sprucewood was hydrolyzed successively with water and dilute sulphuric acid and the hydrolyzates analyzed quantitatively for simple sugars. Four different samples of sulphite waste liquor were each passed through cation and anion exchangers, neutralized with barium hydroxide, and precipitated from 80% alcohol. The sugars remaining in the alcoholic filtrate were likewise analyzed for simple sugars. Compression wood and normal wood from the same log were both cooked by the sulphite process and also hydrolyzed with 0.01 *N* sulphuric acid. The resulting hydrolyzates were analyzed for simple sugars after purification. The results show that the easily hydrolyzable part of the wood polyoses does not contain glucan, or only to a very slight extent, whereas large quantities of glucan occur in the wood polyoses which are difficult to hydrolyze. Araban constitutes 0.5 to 1% of the wood; it is the most readily hydrolyzable polysaccharide. About half the sugar content of sulphite waste liquors of various kinds consist of mannose. Glucose had been definitely detected in rayon waste liquors only. Compression wood of spruce may contain as much as 10% galactan.

Samuelson, O., and Westlin, A., *Svensk Papperstidn.* 51: 179–185 (1948); *C. A.* 43: 398 (1949).
By methods and data previously published, it is shown that the content of thiosulphate and polythionate in sulphite waste liquor is low. Since it is known that these sulphur compounds are formed in appreciable quantities during the sulphite cooking process, the authors assume that the inorganic sulphur compounds are converted into organic nonsulphonic sulphur compounds, combined with lignin. Isolated fractions of ligninsulphonate from different types of sulphite waste liquor were converted to free ligninsulphonic acid by cation exchange and electrometrically titrated. The equivalence point calculated from the total sulphur content was always higher than that found by titration of the sulphonic groups. When barium salts of such ligninsulphonate fractions are heated at 150° in alkaline solution, thiosulphate is formed. It was also shown that a considerable quantity of nonsulphonic sulphur is combined with ligninsulphonic acid which had been heated at an elevated temperature with sulphur or thiosulphuric acid.

Schwabe, K., and Hahn, E.. *Papier-Fabr. Wochbl. Papierfabr.* 1944, 351–353; *C. A.* 43: 401 (1949).
A method is described for the determination of reducing substances in sulphite waste liquors and prehydrolyzates.

Salvesen, J. R., and Hogan, D., *Anal. Chem.* 20: 909–911 (1948); *C. A.* 43: 1182 (1949).
A method is described for determining solids in sulphite waste liquor by neutralizing the sample prior to drying with a known amount of standard NaOH which is deducted from the dry solids value. Loss of volatile organic acids and SO_2 compounds is thus avoided. A procedure is described for wet oxidation with nitric-perchloric acid of liquid or solid samples of sulphite waste liquor and ligninsulphonates. All organic matter can thus be removed conveniently and the oxidized sample is used for determination of total sulphur, silica, and cations in the original sample. The precision for the total sulphur determination is shown to be equal to that obtained by the Carius method.

Samuelson, O., *Svensk Kem. Tids.* 60: 128–130 (1948); cf. *C. A.* 43: 398 (1949); *C. A.* 43: 1183 (1949).
Potentiometric and conductometric titrations of ligninsulphonic acid prepared by ion exchange treatment indicated an excess of bound sulphur. The formation of organic sulphur

compounds during sulphite cooking is not limited to sulphonic acid groups. There is an appreciable amount of carboxyl groups present in ligninsulphonic acid.

MAEDA, H., *J. Soc. Chem. Ind. Japan* 45: 267–272 (1942); *C. A.* 43: 1972 (1949).
The surface and boundary activity of sodium ligninsulphonate was studied by measuring the surface tension and the boundary tension between a dilute aqueous solution (0.1 to 0.5%) and benzene with a de Nouy tensiometer. A marked decrease in surface and boundary tensions was observed with the solution as compared with pure water. The decrease in the two values became greater with the sulphite cooking period until it reached a maximum at 8 hours. The sodium salt of the ethyl derivative had a greater power of suppressing the surface and boundary tensions than the sodium salt of the corresponding ligninsulphonic acid.

ABRAHAMSON, B., LINDGREN, B. O., AND HÄGGLUND, E., *Svensk Papperstidn.* 51: 471–474 (1948); *C. A.* 43: 2428 (1949).
Extracted sprucewood meal was digested with a mixture of sodium bisulphite-sodium sulphite at pH = 5.25. About 33% of the lignin was removed leaving a wood residue with a MeO/S ratio of 2.71. The presence of large amounts of sulphite and sulphate was found to lower the methoxyl values as determined by the Vieboch and Schwappach method. The ligninsulphonic acid formed had an indicated degree of sulphonation corresponding to MeO/S = 2 and a comparatively low molecular weight; it was partly retained by an anion exchanger, most of it dialyzed through cellophane, and only a minor part of it could be precipitated by bis-dimethylaminodiphenylmethane. By hydrolyzing with dilute sulphuric acid, a carbohydrate component was split off from the dissolved ligninsulphonic acid; this was accomplished also by digesting with sulphite cooking acid. In this latter treatment the sulphonation proceeded to give a ratio of MeO/S = 1. It is suggested that this further sulphonation and the splitting off of carbohydrate are interdependent. The fact that the second sulphonation does not change the ultraviolet absorption of the substance shows that sulphonation is not accompanied by a deep-seated change in the structure of lignin.

MAEDA, H., AND KOBAYASHI, K., *J. Soc. Chem. Ind. Japan* 45: 419–425 (1942); *C. A.* 43: 2433 (1949).
The difficulty of cooking larch heartwood is not due to the hindrance of the penetration of cooking liquor into the heartwood tissue by the presence of resin, but is due rather to the formation of some insoluble compound by lignin or ligninsulphonate with resin, as was already pointed out by Erdtman (*C. A.* 33: 7098) in connection with red pine heartwood. By performing experiments of cooking heartwood in two stages, starting with the natural heartwood, extracting the resin after 1 to 6 hours, then resuming the cooking, it was shown that the combination of resin with lignin or ligninsulphonate takes place about 2 hours from the start of the cook.

MAEDA, H., AND KOBAYASHI, K., *J. Soc. Chem. Ind. Japan* 45: 425 (1942); *C. A.* 43: 2433 (1949).
Experiments were carried out to show that the free carbonyl or acidic radical is not present in natural lignin, and that they are produced by heating lignin with dilute acid or alkali. Further experiments were also carried out to show that only insoluble sulphonate is produced in the early stage of sulphite cooking, that by such sulphonation there are produced a sulphonic radical and a phenolic hydroxyl radical, and that with prolonged cooking, i.e., with increasing sulphonation and hydrolysis, lignin begins to go into solution in the form of ligninsulphonic acid. Lignin was rendered totally resistant to sulphonation by treating the original wood with an excess of phloroglucinol in the presence of hydrochloric acid.

UCHIDA, J., *J. Soc. Chem. Ind. Japan* 44: 1072–1078 (1941); cf. *J. Soc. Chem. Ind. Japan* 44: 346 (1941); *C. A.* 43: 2434 (1949).
Methods of determining the amount of methoxyl groups were examined and it was decided that the pyridine method (*C. A.* 8: 2163), with eosin as an indicator, is the most suitable. Mixed chips of spruce and fir were cooked under three different conditions of the composition of cooking liquor, temperature, and time, and the methoxyl content of the original wood, pulp, and the waste liquor were determined and compared. The amount of methoxyl found in the pulp and the waste liquor is 88 to 96% of the total amount contained in the original wood in the case of softwood. Methyl alcohol produced during the sulphite cooking was inferred to have been contained in the wood as an ester of uronic acid. The methoxyl remaining in the lignin amounted to 89 to 96% of the total methoxyl content of the softwoods, and 75 to 80% of the hardwoods.

AULIN-ERDTMAN, G., *Tappi* 32: 160–166 (1949); *C. A.* 43: 4461 (1949).
A review of the ultraviolet spectroscopy of lignin and lignin derivatives.

V. WACEK, A., AND KRATZL, K., *J. Polymer Sci.* 3: 539–548 (1948); *C. A.* 43: 4461 (1949).
When only 1 aliphatic hydroxyl group is present in the side chain, it can be exchanged for a sulpho group if it is in sufficient proximity to the nucleus; however, this exchange occurs only to a limited degree and is not the main reaction of the sulphite process. Ketols with carbonyl and hydroxyl groups in the 1,2 position are not sulphonated and are regarded as acyloins (benzoins). Ketols with carbonyl and hydroxyl groups in the 1,3 position can be converted into sulphonic acids with the sulpho group in place of the original hydroxyl group and are regarded as "aldols." They are converted readily by loss of water into systems of conjugated double bonds which can be easily sulphonated. The "aldol" type compound can be conceived as a system of conjugated double bonds with a "masked" second double bond. Pressure oxidation indicates that the presence of a free phenolic hydroxyl group para to a side chain favors oxidation of the side chain into the aldehyde or carboxyl group. The extent of oxidation varies depending on the side-chain structure, but even a saturated aliphatic chain with no substitute is oxidized to a limited extent. Etherification of the the *p*-phenolic hydroxyl group abolishes its influence on the oxidation of the side chain. In the etherified phenol, as in substances with an unsubstituted nucleus, oxidation of the side chain to an aldehyde or acid is restricted to specific groupings. This applies particularly to substances in which the carbon atom near the nucleus contains carbonyl, hydroxyl, or sulpho group as a substituent. The oxidation process is presumably initiated at the terminal group of the chain and leads, via intermediate formation of the α-keto acid and decarboxylation, to the aldehyde which in turn is converted into the acid, this step occurring with particular ease if the aldehyde is capable of a Cannizzaro reaction. Cleavage to an aldehyde with hot alkali in the absence of air is specific for the "aldol type" grouping in the side chain. This is a reverse aldol condensation and it has been demonstrated that acetaldehyde can be isolated from such degradations. It is suggested that there is an "aldol" configuration in the side chain of a ligroin fraction, especially in a fraction of the ligninsulphonates of sulphite liquor. The latter include, in addition to derivatives of the coniferyl class, derivatives of the type $RCOCH_2CH_2OH$ and $RCOCH_2CH_2SO_3H$, which, e.g., represent stabilized end products of the sulphite process.

LAGERSTRÖM, O., SAMUELSON, O., AND SCHOLANDER, A., *Svensk Papperstidn.* 52: 113–115 (1949); *C. A.* 43: 4851 (1949).
A colorimetric method for the determination of silica in sulphite waste liquor is described.

HEDLUND, I., *Svensk Papperstidn.* 50, No. 11B: 109–110 (1947); *C. A.* 43: 8675 (1949).
Extracted sprucewood meal was made to react with sulphite cooking liquor at 128° for 2 and 5 hours, and then cooked for various lengths of time at 120° with acid-sodium phosphate buffer solution or dilute phosphoric acid. The resulting samples were analyzed for sulphur, methoxyl, and Roe chlorine number. It is concluded that unsulphonated lignin is condensed more readily by acid catalysts. Undissolved, partially sulphonated lignin catalyzes lignin condensation when cooked with free phosphoric acid but the sodium in the sodium phosphate buffered solution counteracts such condensation because sodium ligninsulphonate forms through base exchange.

MARKHAM, A. E., PENISTON, Q. P., AND MCCARTHY, J. L., *J. Am. Chem. Soc.* 71: 3599–3601 (1949); *C. A.* 44: 325 (1950).
A ligninsulphonate preparation purified by dialysis, precipitated as a barium salt from aqueous solution by ethyl alcohol, yields a number of fractions characterized by chemical analysis, ultraviolet extinction coefficients, and diffusion coefficients. When the results of the chemical analyses are calculated to an anhydrous sulphur and ash-free basis, the fractions are of nearly uniform composition. Diffusion coefficients of the several fractions increase progressively in order of precipitation and indicate a corresponding decrease in molecular weight over a wide range. The uniformity of chemical composition and of ultraviolet absorption spectrum and the regular trend in diffusion coefficients indicate the polymeric character of ligninsulphonic acids.

KRATZL, K., *Monatsh.* 80: 314–315 (1949); *C. A.* 44: 325 (1950).
Alkaline cleavage of ligninsulphonic acid gives formaldehyde, as well as vanillin and acetaldehyde. The formaldehyde originates from the side chains in the acid but the mechanism of its formation is not known.

KRATZL, K., HECH-BLECHMANN, AND OSTERBERGER, K., *Monatsh.* 80: 271–287 (1949); *C. A.* 44: 325 (1950).
Since spruce ligninsulphonic acid, when treated with bromine, yields bromoform, model substances were tested for their be-

havior with bromine. The bromoform originates from tribromacetaldehyde but in all cases dibromoacetaldehyde is the major reaction product. Bromination of ligninsulphonic acid yields quantities of these two aldehydes which correspond approximately to quantitative cleavage of the side chains. Chlorination under similar conditions gives dichloroacetaldehyde. The formation of haloforms also takes place with compounds which originally contained no acetyl groups. The scission of the side chain with the formation of dibromoacetaldehyde occurs even before the bromination to tribromoacetaldehyde is complete. The yields of bromoform, dibromoacetaldehyde, and tribromoacetaldehyde obtained on bromination of sodium α-propioguaiacone sulphonate, α-bromopropioguaiacone, propioguaiacone, sodium 1-phenylpropane sulphonate, cinnamaldehyde, α-bromocinnamaldehyde, and sodium ligninsulphonate, respectively, are given.

Kobe, K. A., and McCormack, E. J., *Ind. Eng. Chem.* 41: 2847–2848 (1949); *C. A.* 44: 1698 (1950).
Data are given on the viscosity of four samples each of sulphite waste liquor and sulphate and soda black liquors, the solids contents ranging from 11 to 52%. A plot of the logarithm of the η against temperature on a modified logarithmic scale gives straight lines. The data indicate changing characteristics of the liquors at 30% solids, above which the η rises rapidly.

Freudenberg, K., *Sitzber. heidelberger Akad. Wiss., Math.-naturw. Klasse* 1949, No. 5: 151–158; *C. A.* 44: 2235 (1950).
Coniferyl alcohol is exposed in aqueous solution at pH 7 under lively aeration at 20° for 1 to 3 days to dehydrase isolated from the mushroom *Psalliota campestris* to give a 90% yield of the dehydropolymerizate of coniferyl alcohol. It is purified by dissolving in moist acetone, filtering from the enzyme protein, and precipitating with water. It contains 11.3% acetylatable hydroxyl and exhibits the same properties as native spruce lignin, e.g., the same color reactions with aniline salts, phloroglucinol-hydrochloric acid, etc.; it is cream colored and optically inactive, it yields 1.3% formaldehyde on distillation with H_2SO_4 and has a molecular weight of 820; it gives 95% Klason lignin with 72% H_2SO_4 and dissolves completely in bisulphite, giving a sulphonic acid whose S content and ultraviolet absorption are similar to those of other ligninsulphonic acids; it reacts with thioglycolic acid to give a product containing 15.5% sulphur and which is soluble in Na_2CO_3 solution; methylation with diazomethane and then with dimethyl sulphate gives a product with 33% methoxyl; it is soluble in dilute alkali, alcohols, acetone, and dioxan, but not in Na_2CO_3 solution, ether, and water. It is soluble in 30% sodium *m*-xylenesulphonate but separates on dilution; when it is subjected to the reactions employed during the isolation of cuproxam lignin, it gives a product resembling cuproxam lignin. Polymerization of coniferyl alcohol with acid gives a polymer having the same composition as coniferyl alcohol but differing from the dehydropolymerizate in chemical and physical properties.

Yorston, F. H., *Pulp Paper Mag. Can.* 50, No. 12: 108–111 (1949); *C. A.* 44: 4247 (1950).
Sulphite waste liquor was de-ashed with cation-exchange resin Amberlite IR-120. The de-ashed liquor was passed through columns containing Amberlite IR-4B and IRA-400. The resins reduce dissolved solids to 70 to 80%, acidity to 40%, total carbon to 80 to 90%, and methoxyl to about 95% of original values. Reducing sugars were 90 to 95% rejected by Amberlite IR-4B and absorbed variably by IRA-400. Variables such as flow rate, sample dilution, and particle size of resin were studied.

Yorston, F. H., *Pulp Paper Mag. Can.* 50, No. 12: 111–113 (1949); *C. A.* 44: 4247 (1950).
Sulphite waste liquor was de-limed by Amberlite IR-120, passed through Amberlite IR-4B which does not remove sugars, the pH was adjusted with ammonium hydroxide, and the liquor passed through Amberlite IRA-400 which absorbs sugars. The sugars were recovered by saturating the exchanger with carbon dioxide, in a recovery of 60 to 70%. The resulting solution, after concentration, yielded the following: white crystals (0.85 gram) containing 93% pentose (xylose identified by the double salt of cadmium xylonate and cadmium bromide) brown crystals (0.76 gram) containing 80% pentose, and syrup (5.83 gram) containing 49% pentose.

Ritter, D. M., Olleman, E. D., Pennington, D. E., and Wright. K. A., *J. Am. Chem. Soc.* 72: 1347–1351 (1949); *C. A.* 44: 4674 (1950).
A specially purified ligninsulphonate which fractional diffusion analysis had shown to be chemically homogeneous yielded a hemimethoxy derivative with diazomethane, a permethoxy derivative with dimethyl sulphate and alkali, and a water-soluble derivative when cautiously acetylated with sodium acetate as a catalyst. The methyl esters of the three derivatives were prepared by the action of diazomethane upon the carefully dehydrated free acids. Systematic and comprehensive analyses of the ligninsulphonate derivatives in the form of the ammonium or barium salts or of the methyl esters suggest that all the derivatives originated from a ligninsulphonic acid with an average empirical formula $C_9H_{7.5}(OH)(OMe)(SO_3H)_{0.5}$.

Braun, K., and Grause, G., *Das Papier* 4: 44–48 (1950); *C. A.* 44: 4677 (1950).
A rapid method has been devised for the determination of volatile organic acids (calculated as acetic acid) in sulphite waste liquor. The liquor (10 ml.) is treated with sufficient 0.1 *N* (or *N*) chloramine to oxidize the SO_2 in solution. The treated liquor, in a distilling flask connected with a condenser dipping into a receiving flask containing 20 ml. (CO_2-free) distilled water in a glycerol bath at 130 to 140°, is distilled to dryness and thereafter kept at 130° for 5 minutes. After cooling, 10 ml. distilled water is added to the dry residue and the mixture redistilled to dryness and reheated at 130°. The combined distillate is heated with 5 ml. KI-starch solution and accurately titrated with chloramine to a pale blue color (discharged by 1 drop 0.1 *N* $Na_2S_2O_3$); 2 drops Phenol red is then added and the mixture titrated to a red end point with 0.1 *N* NaOH. The difference between the final titration and that with chloramine is a measure of the organic acid (calculated as acetic). The ether extraction method of Doering (*C. A.* 38: 4128) was also studied. With slight modifications (i.e., exhaustive extraction and prevention of emulsions) it gave excellent results. Results by the distillation and extraction methods agreed well, unless the original liquor was extremely dark (e.g., in the production of soft pulps). In such cases the distillation method gave values that were much too high because of the secondary formation of volatile organic acids not originally present in the liquor. This was obviated when the original liquor was titrated with aqueous $Ba(OH)_2$ to pH 2.3 prior to distillation.

Erdtman, H., Lindgren, B. O., and Pettersson, T., *Acta Chem. Scand.* 4: 228–238 (1950); *C. A.* 44: 7530 (1950).
Ligninsulphonic acid of a low degree of sulphonation was further sulphonated by heating under pressure with sulphite cooking acid for 1¼, 2, and 10 hours. Three ligninsulphonic acids of higher degrees of sulphonation were isolated. The barium salts of the four ligninsulphonic acids were analyzed for methoxyl, sulphur, barium, carbon, and hydrogen. The pyridinium salts of the corresponding four acetylated ligninsulphonic acids were analyzed for methoxyl, sulphur, nitrogen, carbon, hydrogen, and acetyl. The data show that the entry of each new sulphonic acid group into the lignin was accompanied by the loss of an hydroxyl group. The low sulphonated ligninsulphonic acid contained 1 S per 3 C_{10} elements; the highest sulphonated contained 1 S per 1.4 C_{10} elements. These results are compared with the work of Freudenberg, Lautsch, and Piazolo (*C. A.* 40: 3258) Holmberg (*C. A.* 37: 6451), and Peniston and McCarthy (*C. A.* 42: 4745).

Central Laboratory of the Swedish Cellulose Industry, Analysis Committee, *Svensk Papperstidn.* 52: 597–598 (1949); *C. A.* 44: 7536 (1950).
A standard method for the determination of sulphate in sulphite waste liquor based on previous work is described.

Kratzl, K., and Rettenbacher, F., *Monatsh.* 80: 622–633 (1949); *Osterr. Akad. Wiss. Math.-naturw. Klasse, Sitzber. Abt. IIb* 158: 622–633; *C. A.* 44: 8637 (1950).
In an atmosphere of nitrogen, ligninsulphonic acids and model compounds were hydrolyzed by boiling 1 *N* NaOH. Volatile aldehydes were determined in the distillate by precipitation with 2,4-dinitrophenylhydrazine or *m*-nitrobenzhydrazide. Nonvolatile decomposition products should be isolated from the hydrolysis liquors. When 4,3-HO(MeO)C_6H_3COCH: $CHC_6H_3(OMe)_2$-3.4 or its sulphonic acid was hydrolyzed, veratraldehyde was collected in the distillate (70–90% of theoretical in 6 hours) and acetovanillone was found in the alkaline liquor. In the distillate from the hydrolysis of flavanone and of cinnamaldehyde sulphonic acid, benzaldehyde was determined (90% theoretical in 3½ hours), and, in the latter case, about 20% of the theoretical amount of acetaldehyde. These carbonyl compounds apparently are formed by a hydrolysis of the parent substance which is the reverse of the aldol condensation. When spruce ligninsulphonic acid was hydrolyzed, acetaldehyde was collected in the distillate and vanillin was found in the alkaline liquor. Thus, both decomposition products of coniferylaldehyde, which has been proposed by Freudenberg and others as a structural unit in lignin, have been isolated from ligninsulphonic acid in approximately equimolar quantities accounting for 2 to 3% of the lignin. Stepwise hydrolysis showed that vanillin was not formed as continuously as acetaldehyde. From beech ligninsulphonic acid, subjected to alkaline hydrolysis acetaldehyde, vanillin, and syringaldehyde have been isolated'

Enkvist, T., and Hägglund, E., *Svensk Papperstidn.* 53: 85–93 (1950); *C. A.* 44: 8641 (1950).
Methylation of sprucewood with dimethyl sulphate and NaOH at room temperature almost completely inhibits the formation of alcohol–soluble thiolignin when heated at 100° and pH 7

with H_2S or with NaHS and Na_2S at 160°. However, sulphur is bound in the solid alcohol-insoluble wood by these treatments. Methanol lignin and wood treated with MeOH-HCl also took up considerably less sulphur by heating with H_2S at pH 7 as compared with similar sulphidization of HCl lignin, cuproxam lignin, alkali lignin, and native lignin. Ligninsulphonic acids with different degrees of sulphonation, when sulphidized with H_2S at pH 7 at 100°, took up only 1–2% S and the sulphonic groups remained practically unchanged. However, they were split off when the ligninsulphonates were cooked at 160° with NaHS solution and gave mainly a thiolignin with 12% S. Wood which first was sulphidized with H_2S at pH 7 at 100° and cooked with regular sulphite cooking acid was strongly sulphonated but the ligninsulphonate was not dissolved from the wood. When sulphidized wood was first cooked with $NaHSO_3$ solution at pH 5.5 and then with a solution of $NaHSO_3$ and SO_2, the lignin became water soluble and there resulted a normal pulp with low lignin content. These experiments indicate that the same groups in lignin react on sulphidization as on sulphonation and in the formation of alcohol lignins. In discussing the possible mechanism of the sulphidization in light of the present and previously reported findings, it is pointed out that phenolic groups in thiolignin may prevent the solution of ligninsulphonates after digestion with regular cooking acid, but permit it by the two-stage sulphite process—in analogy to sulphite cooking of pine wood.

15. MISCELLANEOUS USES

Gössel, F., Ger. pat. 722,691 (June 4, 1942); *C. A.* 37: 5160 (1943).
Fresh or concentrated sulphite waste liquor is added to silos to stabilize the vegetable matter.

Pritchard, W. C., and Daily, R. A., U. S. pat. 2,325,542 (July 27, 1943); *C. A.* 38: 487 (1944).
Ligninsulphonic acid suitable for use as an addition agent to storage-battery negative paste (for giving the plates increased life and capacity, especially at low temperatures) is obtained by treating sulphite waste liquor with sulphuric acid to bring the specific gravity to 1.3. The precipitate which contains ligninsulphonic acid is separated from the remainder of the liquor and the lignin leached out with water.

v. Engelhardt, W., *Oel u. Kohle* 39: 707–709 (1943); *C. A.* 38: 1045 (1944).
In order to increase the penetration rate in drilling through formations containing hard quartz, experiments were carried out in which quartz crystals were ground with silicon carbide powder in the presence of various liquids. Aqueous solutions of polar liquids and electrolytes are slightly effective hardness reducers and require high concentrations of additive. Salts of fatty acids are relatively more effective and aqueous solutions of sulphite waste liquor are satisfactory hardness reducers.

Racky, G., Ger. pat. 713,866 (Oct. 23, 1941); *C. A.* 38: 1878 (1944).
Concentrated or dry sulphite liquor is treated with strong fluosilicic acid with vigorous stirring in the presence of an alcohol or phenol insoluble in the acid.

Rieche, A., and Rudolph, W., Ger. pat. 715,322 (Nov. 27, 1941); *C. A.* 38: 2490 (1944).
Ligninsulphonic acid is converted into calcium ligninsulphonate. The salt is separated from the liquor, then caused to react with a zirconium salt, the anion of which precipitates calcium.

Elöd, E., and v. Schachowskoy, T., Ger. pat. 736,957 (May 20, 1943); *C. A.* 38: 3840 (1944).
Chromium compounds capable of forming chromic hydroxide are added to purified sulphite liquor or its extract. For each mole of the sulpho group of the ligninsulphonic acid is added one mole of chromium. The pH is kept at 2.3 to 4.0.

Baranov, V. S., *Neftyanaya Prom.* 22, No. 6: 64–69 (1941); *Chem. Zentr.* 1943, II: 978; *C. A.* 38: 5385 (1944).
A discussion of the preparation of fresh clay suspensions with brown coal and alkali, and the addition of material for increasing the specific gravity, as well as the maintenance of a suitable condition of the clay suspension with respect to the strata which are being drilled. Tests were made of the involved method of Baranov and Buks (*C. A.* 31: 2804) for the treatment of clay suspensions with brown coal, sulphite liquor, and alkali.

Wilson, H. D., U. S. pat. 2,367,453 (Jan. 16, 1945); *C. A.* 39: 2936 (1945).
A process is described for the utilization of sulphite waste liquor in the manufacture of storage battery plates. The sulphite waste liquor is neutralized with lime and an excess added to bring the pH to 9.6. The precipitated calcium sulphite is removed and the supernatant liquor is concentrated by evaporation to approximately 47% solids. Excess ammonium hydroxide is added and the whole heated to 173°F. under approximately 140 p.s.i. pressure for $1^1/_2$ hours. The solids are thereafter separated from the liquor and dried to a paste. When treating sulphite waste liquor from hemlock pulp, the fixed nitrogen content amounts to 3%. When incorporating it into the active material of the plate, 0.1 to 3.0% by weight of the above product is used. In addition other expanders are utilized, e.g., barium sulphate and/or lampblack, and mixed to a paste with lead oxide, which is subsequently applied to the storage battery plate in the usual manner. The above ammoniated substance greatly improves the performance of storage batteries at low temperatures.

McFarlane, W. D., and Nikolaiczuk, N., U. S. pat. 2,418,311 (Apr. 1, 1947); *C. A.* 41: 3884 (1947).
A protein-fat concentrate suitable for use in mixed feeds may be prepared from chick-hatchery waste by cooking it with sulphite waste liquor. The coagulum, after pressing and drying, contained on a shell-free basis: crude protein 50.35, fat 39.75, ash 2.46, and water 1.2%. The amounts of fat and protein recovered were approximately 92 and 94%, respectively.

Hull, R. O., and Gray, A. G., Brit. pat. 583,003 (Dec. 4, 1946); *C. A.* 41: 6477 (1947).
At high current densities, the operation of acid copper plating baths is enhanced by the addition of a mixture consisting of benzoquinone and sulphite waste liquor together with an antifoaming agent.

Budnikov, P. P., and Butt, Y. M., *Compt. rend. acad. sci. U.R.S.S.* 56: 503–505 (1947); *Chem. Zentr.* 1947, I: 846 *C. A.* 42: 9176 (1948).
Sulphite waste liquor was added to gypsum-hemihydrate in amount of 0 to 0.5%. The addition reduced the water demand of the gypsum, increased its hardness, and had no effect on the setting period.

Dunbar, T. L., U. S. pat. 2,470,764 (May 24, 1949); *C. A.* 43: 5950 (1949).
Sulphite waste liquor is treated with an oxidizing agent, such as hydrogen peroxide or calcium hypochlorite, and the resultant sludge is separated from the liquor, preferably with the addition of filter aid or sawdust. The solution is treated with sodium chloride to salt out the lignin. The precipitate is absorbed on unbleached pulp, such pulp thus increasing 15 to 20% in weight. The clarified liquor is mixed with fresh liquor in the ratio of 3 to 1, brought up to required strength in free and combined sulphur dioxide and is used for digestion of a fresh batch of chips.

Imaizumi, M., Japan pat. 174,469 (Feb. 3, 1947); *C. A.* 44: 840 (1950).
The color is removed from sulphite waste liquor by mixing with less than 10% TiO_2.

Roschier, H., *Suomen Kemistilehti* 23A: 93–95 (1950); *C. A.* 44: 11095 (1950).
Previous experiments have shown it to be possible to extract sugar from wood chips by short heating with sulphite waste liquor (*C. A.* 38: 2486). Chips thus pretreated cannot be successfully pulped by the sulphite process because the lignin present has been made insoluble by the pretreatment. A pulp containing up to 95% α-cellulose and of low pentosan content can be prepared from such chips by the sulphate process.

16. POLLUTION STUDIES

Tyler, R. G., *Sewage Works J.* 14: 834–838 (1942); *C. A.* 39: 4707 (1945).
The difficulty of treating sulphite waste liquor as compared with domestic sewage is examined. A method is proposed in which the dissolved oxygen content of the receiving water is brought up to any desired requirement by artificial aeration, after the natural biological processes have been allowed to take place.

Holderby, J. M., *Sewage Works J.* 18: 671–677 (1946); *C. A.* 41: 1838 (1947).
Laboratory trickling filters were constructed 1 foot square and 5 feet deep, with 1-inch ceramic Raschig rings as filter medium. One filter was equipped for recirculation of effluent. Sulphite liquor diluted with sewage or river water to B.O.D. values up to 3730 p.p.m. has been successfully treated to remove better than 75% of the applied B.O.D. at loading in excess of 6 pounds per cubic yard per day. The Raschig rings were not satisfactory because they did not slough easily. Nitrogen and phosphorus must be added, but inorganic forms are satisfactory. Recirculation of effluent indicated some improvement. Biological forms in slime were mostly molds and yeasts with an almost complete absence of protozoans.

Tyler, R. G., Maske, W., and Brewer, R., *Sewage Works J.* 18: 1155–1168 (1946); *C. A.* 41: 1839 (1947).
The B.O.D. of sulphite waste liquor is due largely to the immediate demand of sulphur dioxide, sugars, and acetic acid, the lignin being so slowly oxidized that it is negligible. The inhibiting effect of sulphur dioxide is eliminated by steam stripping, which raises the pH from 1.5 to 3.5. The addition of nutrient ammonia still further raises the pH to 4.0. This liquor as such and liquor buffered to pH 7.0 were treated on lab-scale filters filled with crushed stone. The organisms which develop and produce the purification are yeasts, molds, and various acclimated bacteria. At B.O.D. loadings of 1.5 to 5 pounds per cubic yard 85 to 90% removals were obtained. As the loadings increased to 25 pounds B.O.D. per cubic yard, the removals decrease in a straight line function to about 50%. Recirculation aided in preventing clogging from molds but did not increase filter loadings. The clogging of filters is due to mold growths. Phosphorus is not essential as a nutrient but nitrogen must be used.

Warrick, L. F., *Ind. Eng. Chem.* 39: 670–676 (1947); *C. A.* 41: 3965 (1947).
A brief historical résumé of industrial wastes in the pulp and paper industry which shows the trend from individual mill studies through state or regional programs to the more recent country-wide activities of the industry to reduce, utilize, or treat wastes to minimize water pollution. Chemical pulping wastes, such as sulphite waste liquor, are the strongest from a pollutional point of view, and the most difficult to utilize or treat and dispose of in a practical and economical manner. In general, costs for taking care of wastes to prevent serious stream pollution are increasingly to be recognized as an essential part of production costs.

Logan, R. P., and Heukelekian, H., *Sewage Works J.* 20: 282–291 (1948); *C. A.* 42: 9176 (1948).
Liquid-phase oxidation of sulphite waste liquor consisted in aeration with the organisms in suspension and at a rate low enough to control foaming. With an acclimated growth of dispersed mixed culture cells the B.O.D. was reduced 30% in 24 hours. If the sulphite liquor is shaken in an oxidation apparatus with pure oxygen, the reduction was 35%. Use of yeast cells did not improve the results. Foam-phase treatment consisted in aeration at rates to convert all the liquid into a foam phase. In this case a tan color slime growth built up on the side walls and reduced the B.O.D. 80 to 85% in 24 hours. Yeast cells gave only 50 to 60% reductions. Continuous operation gave similar results to the 24-hour batch operation.

Tyler, R. G., and Gunter, S., *Sewage Works J.* 20: 709–719 (1948); *C. A.* 43: 3126 (1949).
Details are given for determining B.O.D. in liquor containing sulphur dioxide, toxic materials, pentoses, hexoses, lignin, and organic acids.

Tully, J. B., *Paper Trade J.* 129, No. 4: 35–40 (1949); *C. A.* 43: 7684 (1949).
The factors influencing stream pollution by a sulphite mill are B.O.D. and quantity of sulphite waste liquor, quantity of total mill effluent, dissolved oxygen, and total flow of stream into which the waste is dumped. These factors have been related with formulas enabling the calculation of the degree of pollution (P), which is defined in general terms as the pollution effects/limit of tolerance, with tolerable pollution having a $P = 1$ or less. Detailed examples are given of the calculations for a fresh water stream and a salt water tidal inlet.

Chase, E. S., *J. Boston Soc. Civil Engrs.* 36: 357–382 (1949); *C. A.* 43: 7614 (1949).
The tremendous pollutional effect of sulphite waste liquors is compared with all other sources of pollution. There is an apparent benefit in odor control by the addition of sodium nitrate to the river.

Odlaug, T. O., *Trans. Am. Microscop. Soc.* 58: 163–182 (1949); *C. A.* 43: 8136 (1949).
Stabilization of sulphite waste liquor occurs after discharge into sea water, and toxic effects are reduced. The toxicity was measured by the rate of pumping of Olympia oysters, *Ostrea lurida.*

Tully, J. P., *Fisheries Research Board Canada Bull.* No. 83: (1949); *C. A.* 44: 1698 (1950).
A complete study was made to determine the effect of introducing the waste from a 135-ton sulphate mill into Alberni Inlet.

Delcroix, P., et al., *Bull. assoc. tech. ind. papeterie* 3: 73–84 (1949); *C. A.* 44: 2236 (1950).
An informal symposium on industrial waste liquors from the pulp and paper industries of France and their effects on stream pollution.

Holderby, J. M., and Wiley, A. J., *Sewage and Ind. Wastes* 22: 61–70 (1950); *C. A.* 44: 6062 (1950).
Various methods of treatment are discussed and some experimental data are presented on pilot-plant studies of trickling filters, contact aeration, anaerobic methane production, and yeast production. Comparative loadings and efficiencies of known methods are given.

Rumpel, W., *Mitt. chem. Forsch-Inst. Ind. österr.* 4: 6–9 (1950); *C. A.* 44: 9672 (1950).
The problem of disposal of sulphite waste liquor with its inherent loss of by-products and pollution of rivers is discussed, and attempts are described to solve the problem by replacing Ca in the cooking acid by NH_4, Mg, or Na. Whether these attempts offer a permanent solution is questionable, since the elimination of the sulphite waste liquor also eliminates the possibility of recovering valuable by-products, such as alcohol, raw proteins, tanning agent, or lignin. The loss of running the waste liquor into rivers is about 1 ton organic by-products per ton of pulp produced or 50% of the wood treated.

17. REVIEW ARTICLES

Wise, L. E., *Paper Ind. Paper World* 24: 1223–1227 (1943); *C. A.* 37: 3600 (1943).
A brief review dealing with "sulphite liquor lactone," cymene from sulphite terpentine, "pseudo-yeast" (Torula yeast), and solvents (butanol, acetone, and ethyl alcohol) by fermentation of the sugars of sulphite waste liquor by *Clostridium butylicum.*

Wagner, K., *Vierjahresplan* 5: 924–926 (1941); *Chem. Zentr.* 1942, I: 2326; *C. A.* 37: 4244 (1943).
The role played by lignin in the plant and its chemical structure are discussed. Its solution during the sulphite pulping of wood depends on the addition of calcium bisulphite to a double bond and aldehyde group with a simultaneous hydrolytic decomposition of the whole complex. Commercially lignin can be obtained in the saccharification of wood by the Bergius or the Scholler process as a difficultly soluble solid. It also occurs in sulphite waste liquors as ligninsulphonates, together with fermentable sugars, resins, fats, etc. Wood sugar lignin can be used as a fuel without much trouble; however, sulphite waste liquor must be subjected first to a costly evaporation process. By heating calcium ligninsulphonate with sodium hydroxide and adding wood flour it can be converted into synthetic products. Barium ligninsulphonate which can be prepared by treatment with barium oxide is commercially important. Tanning products can be made by precipitating calcium from waste sulphite liquor. Bleached sulphite liquor yields resin soaps which can be converted into useful detergents by mixing with small amounts of potassium or sodium soaps. Vanillin can be obtained by pressure decomposition of calcium ligninsulphonate with sodium hydroxide, or by direct treatment of sulphite waste liquor with sodium hydroxide and oxygen. Approximately 12% of the lignin content of waste sulphite liquor can be broken down by a cracking process to cyclohexanol and cyclopentanol. Sulphite waste liquor can also be converted to adhesives, fertilizer, and wood impregnating agents.

Klingstedt, F. W., *Finnish Paper Timber J.* 22: 441–442 (1940); *C. A.* 37: 4895 (1943).
The manufacture of vanillin and of lignin boards from sulphite waste liquor is briefly discussed. Some properties of such boards are outlined.

Wagner, K., *Wochbl. Papierfabr.* 73: 341–344 (1942); *C. A.* 37: 5232 (1943).
A brief descriptive article which deals with possible uses of lignin (obtained from acid hydrolysis of wood and sulphite waste liquors). Utilization may be direct or by conversion through by-products obtained on heating, alkali fusion, or hydrogenation. The by-products are listed and some of their uses discussed. Reference is made to the Lautsch vanillin process (alkaline oxidation of sulphite waste liquor in the presence of oxygen or nitrobenzene and the Lautsch cracking process in which cyclohexanols and cyclopentanols are obtained from lignin.

Pevtsov, S. I., and Vekhotko, T. I., *Khim. Referat. Zhur.* 4, No. 4: 126 (1941); *C. A.* 37: 5589 (1943).
A short description is given of existing methods for the utilization and purification of sulphite waste liquor, e.g., processes of boiling and utilization of sulphite pulp, chemical methods for precipitation, and chemical and biological methods for the oxidation of the organic constituents of the liquors. It is concluded that neither the methods of chemical oxidation (e.g., with chlorine) nor those of biological purification are satisfactory.

Weidner, J. P., *Paper Mill News* 66, No. 30: 12, 14 (1943); *Pulp Paper Mag. Can.* 44: 739–740 (1943); *C. A.* 37: 6455 (1943).

An outline of the cooperative research program on disposal of sulphite waste liquor fostered by the Wisconsin sulphite mills, with a review of the development of the organization now existing, the nature of the projects undertaken, and their present status.

BRAUNS, O., *Svensk Papperstidn.* 46: 244–253, 271–280 (1943); *C. A.* 37: 6890 (1943).
A review of Swedish developments including recovery of sulphite waste liquor for alcohol manufacture, evaporation of sulphite waste liquor and black liquor and other projects undertaken on cellulose.

SCHWABE, K., *Papier-Fabr.* 41: 37–43, 49–52 (1943); *C. A.* 37: 6890 (1943).
A discussion of the utilization of sulphite waste liquor giving no new experimental data. Delignification reactions and the composition of the liquor are outlined. The utilization of the pentose and hexose sugars in the production of alcohol and feedstuffs is described. The following possible uses of sulphite waste liquor are suggested: tanning agents, core binders, briquet binders, fuel (with special emphasis on the Ramen process) and the formation of hydrogenation products. The limitations of beechwood sulphite waste liquor are mentioned.

LEHMANN, E., *Seifensieder-Ztg.* 69: 13–14 (1942); *C. A.* 38: 252 (1944).
A review on the chemical utilization of sulphite waste liquor.

THIEL, K., *Z. ges. Schiess-u. Sprengstoffw. Nitrocellulose* 38: 104–105 (1943); *C. A.* 38: 2203 (1944).
A review on sulphite waste liquor as a raw material.

MOHLMAN, F. W., *Sewage Works J.* 15: 1164–1181 (1943); *C. A.* 38: 4358 (1944).
In a discussion of industrial wastes in wartime, it is suggested that sulphite pulp wastes might produce by fermentation 16.2 million gallons of alcohol per year, leaving the lignin which might be removed by the Howard process. At Appleton, Wis., the treatment of these wastes is being studied with trickling filters, methane fermentation, contact aeration, and ponding with stream-flow control.

WALDENSTRÖM, E., *Tek. Tid. Kemi* 73: 47–53 (1943); *Chem. Zentr.* 1943, II: 1412; *C. A.* 38: 5397 (1944).
A survey is given of the waste resulting from the cutting, the mechanical and chemical utilization of wood (branches, bark, sawdust) and by-products (lignin, sulphite alcohol, and waste liquors). The possibilities of more complete utilization of wood through the use of these materials are discussed.

BRAUNS, O., *Svensk Papperstidn.* 47: 305–311 (1944); *C. A.* 38: 6554 (1944).
The following Scandinavian developments for 1943 are reviewed; evaporation of sulphite waste liquor; combined evaporation of sulphite and sulphate waste liquor; soda recovery; development of disk evaporator for complete drying of black liquor; Ottersland chipper; chip classifier for control work; apparatus for separating knots from chips; fusel oil from sulphite alcohol; Sandell stiffness tester for paper and board.

LASSENIUS, T., *Finnish Paper Timber J.* 26: 73, 85 (1944); *Tech. Bull. Papermakers Assoc. Gt. Brit. and Ireland* 21: 45; *C. A.* 39: 616 (1945).
The by-products of sulphite mills include cymene (0.2 kg. per ton of pulp). Sulphite waste liquors yield active carbon, fertilizers, synthetic resins, tanning agents, permutites, vanillin, acetic acid, acetone, pyrocatechol, phenols, and alcohols. The theoretical yield of alcohol is 95 kg. of 100% ethanol per ton of pulp and over 160 kg. per ton of rayon pulp, whereas in practice the yields are 75 and 125 kg., respectively. The alcohol by-products amount to 4 to 5 kg. per ton of pulp; 0.5% of the alcohol consists of isobutyl, propyl and heptyl alcohols. Yeast can be prepared from the unfermented liquor, utilizing the hexoses; pentoses can be used also; 2 to 3 kg. per ton of pulp is obtained by the alcohol plants. The by-products of the sulphate mills are also discussed.

PEPPER, J. M., *Pulp Paper Mag. Can.* 46: 83–91 (1945); *C. A.* 39: 1985 (1945).
A review of the isolation and utilization of ligninsulphonic acids (126 references).

GEHM, H. W., *Sewage Works Eng. and Munic. Sanit.* 16: 496–497 (1945); *C. A.* 40: 659 (1946).
A discussion of trends in handling pulp and paper mill wastes. The development of by-products recovery from waste liquor is in the direction of obtaining lignin, vanillin, plastics, and ethanol. The pulping processes employing magnesia, sodium, and ammonium-base liquors in which the organic material constituents are burned for steam production are receiving increased attention. There are also under development processes for evaporation and burning of calcium-base liquors.

YORSTON, F. H., *Pulp Paper Mag. Can.* 47: 131–134 (1946); *C. A.* 40: 4513 (1946).
A review with 43 references of recent advances in the chemistry of wood hydrolysis, cellulose, lignin, waste liquors, and minor constituents of wood.

RUSSELL, J. K., *Pulp Paper Mag. Can.* 47: 277–279 (1946); *C. A.* 40: 4516 (1946).
A review on the utilization of wood wastes covering papermaking materials, oxalic acid, and alcohol (22 references).

HOWROYD, R., *Ind. Fibres, Synthetics and By-products* 7: 238–242 (1945); *C. A.* 40: 5248 (1946).
Liquors from the sulphite and sulphate pulp processes are no longer wasted as they are valuable as a source of various products, e.g., tanning materials. Lignin research, methods of utilization of the organic constituents of the waste liquors, composition of tall oil, and the continued search for other useful products are discussed.

ANON., *Papeterie* 68: 194–195 (1946); *C. A.* 41: 857 (1947).
The production of yeast, vanillin, and tannin auxiliaries from sulphite waste liquor is discussed briefly.

MCGOVERN, J. N., AND DICKERMAN, G. K., *Paper Trade J.*, 124, No. 2: 33–44; No. 3: 43–52 (1947); *C. A.* 41: 1432 (1947).
A general description is given of the manufacture of pulp and paper in Germany, the disposal of sulphite waste liquor, and special paper and board products developed during the war.

KRATZL, K., *Experientia* 2: 469–476 (1946); *C. A.* 41: 1838 (1947).
A review of ligninsulphonic acids and their technical utilization (42 references).

ARIES, R. S., *Northeastern Wood Utilization Council, Bull.* No. 14: 97–123 (1947); *C. A.* 41: 2240 (1947).
A general review of recent developments in the utilization of sulphite waste liquors.

YORSTON, F. H., *Pulp Paper Mag. Can.* 48: 296, 298, 300, 304, 306 (1946); *C. A.* 41: 2889 (1947).
A review of recent advances in the chemistry of wood. It includes wood properties in relation to pulp quality, wood cellulose, and hemicellulose, reactions of cellulose, delignification of wood, lignin and waste liquor, chemical utilization, and the minor constituents of wood.

HILL, A. C., *Can. Chem. Process Ind.* 31: 914–917, 920 (1947); *C. A.* 42: 755 (1948).
A review of Canada's major industry including discussion of mechanical pulp, bleached groundwood, the sulphite process, ligninsulphonic acids, tanning liquors, other uses of waste liquor, sulphite pulps, the sulphate process, tall oils, and the manufacture of kraft products.

MATAGRIN, A., *Papeterie* 68: 296–299 (1946); 69: 10–14 (1947); *C. A.* 42: 3176 (1948).
A detailed review (over 230 references) which includes methods of analysis of waste liquors from pulping, the composition of sulphite and sulphate waste liquors, their utilization, and the recovery and uses of by-products.

BENSON, H. K., *Paper Trade J.* 126, No. 12: 59–62 (1948); *C. A.* 42: 3176 (1948).
A discussion is given of products from sulphite waste liquor (alcohol, lignin, fuel, fertilizer, and yeast) and bark (plywood glues, crop dusting compounds, and core binders from Douglas-fir and fibers for textiles, insulation, and dust for soil conditioning from red wood).

JAHN, E. C., *Paper Mill News* 71, No. 10: 14, 16, 20 (1948); *C. A.* 42: 3959 (1948).
A review of the developments in chemical production from pulp mill by-products in Scandinavia. Chemicals are produced from ethanol (sulphite liquor alcohol) and tall oil (from sulphate liquors). Other products are cymene, lignin, yeast, and cellulose derivatives.

BAUM, M., *Northeastern Wood Utilization Council, Bull.* No. 19: 55–57 (1948); *C. A.* 42: 4745 (1948).
A review of the chemistry and applications of lignin from sulphite liquor.

LLADO, J., *Afinidad* 22: 117–121, 179–183, 210–23 (1945); *C. A.* 42: 5660 (1948).
A recapitulation and brief treatise covering the principal work on lignin during recent years. It includes methods of obtaining lignin, chemical properties, constitution, analysis, reactions, derivatives and industrial applications, with special emphasis on vanillin and plastic products.

LEWIS, H. F., *Paper Trade J.* 127, No. 2: 51–55 (1948); *C. A.* 42: 7040 (1948).
A review of various uses of sulphite waste liquor.

TOMLINSON, G. H., *Pulp Paper Mag. Can.* 49, No. 7: 63–68 (1948); *C. A.* 42: 7040 (1948).
The author reviews his activities in the manufacture of alco-

hol from wood, the soda recovery system, the development of the Babcock Wilcox-Tomlinson recovery furnace, the utilization of sulphite liquor, and the magnesium-base sulphite pulping process.

PEARL, I. A., *Chem. Eng. News* 26: 2950–2953 (1948); *C. A.* 43: 398 (1949).
A review with special emphasis on the utilization of sulphite waste liquor as a raw material for a synthetic organic chemical industry.

KOLASINSKI, R. D., *Wisconsin Engr.* 53, No. 1: 15–17, 30 (1948); *C. A.* 43: 792 (1949).
Types of industrial waste and means of handling them, particularly sulphite waste liquor, are reviewed.

BRAUNS. F. E., *Econ. Botany* 2: 419–435 (1948); *C. A.* 43: 1841 (1949).
A review of the occurrence, chemistry, decomposition products, and uses of lignin.

RAFF, R. A. V., *Can. Chem. Process Inds.* 33, No. 1: 35–40 (1949); *C. A.* 43: 2767 (1949).
A review of recent advances in the uses and knowledge of lignin.

ERDTMAN, H., *Tappi* 32: 71–74 (1949); *C. A.* 43: 2767 (1949).
A review of the chemical nature of lignin.

DAWSON, T. R., *J. Rubber Research* 18: 1–11 (1949); *C. A.* 43: 2768 (1949).
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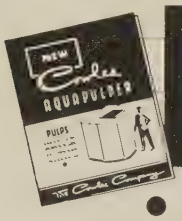

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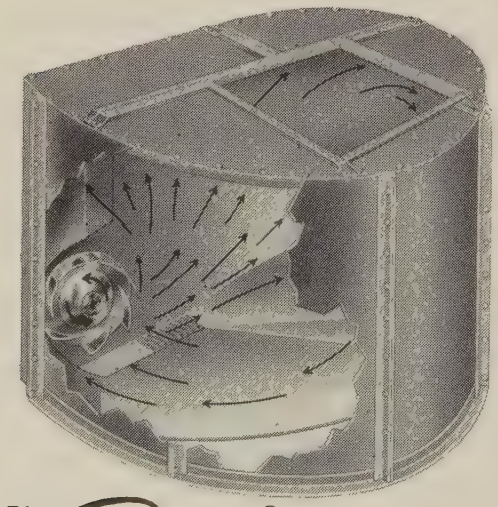

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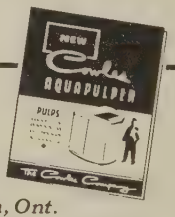

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DELAWARE VALLEY SECTION:
March 27, 1952, Engineers Club, Philadelphia, Pa.

KALAMAZOO VALLEY SECTION:
February 7, 1952, Kalamazoo, Mich.

OHIO SECTION:
February 12, 1952, Middletown, Ohio

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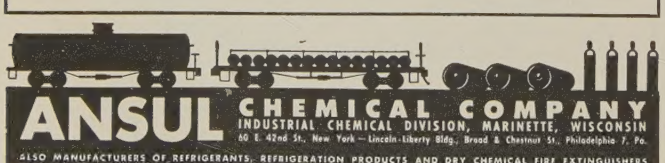

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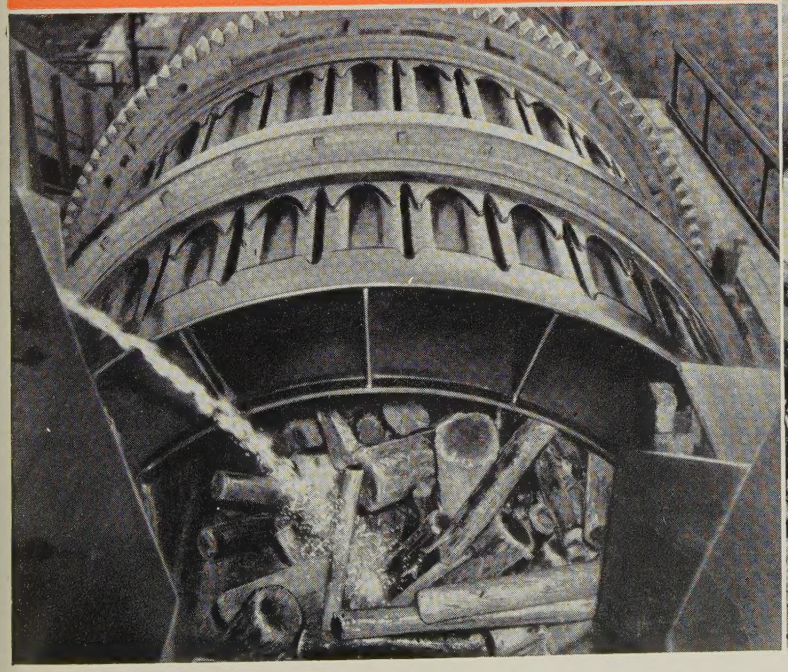

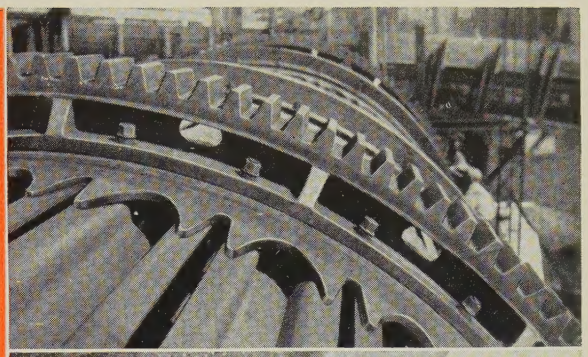

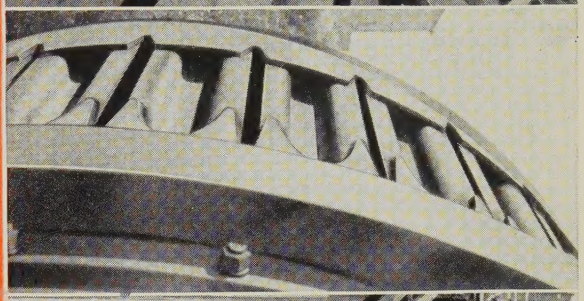

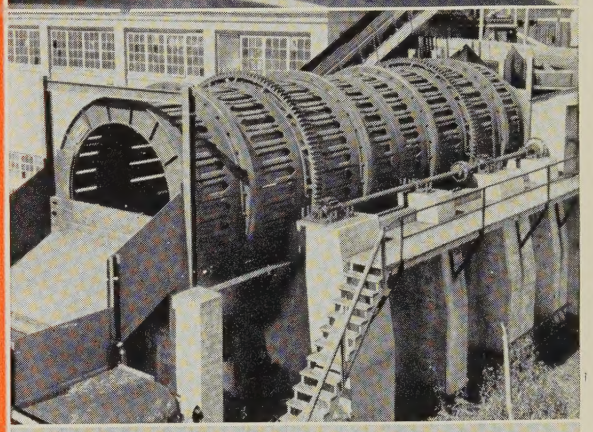

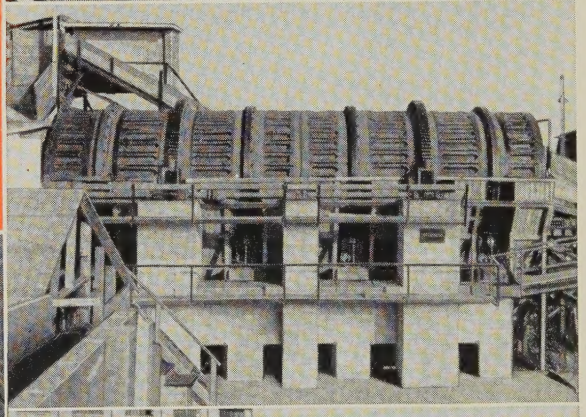

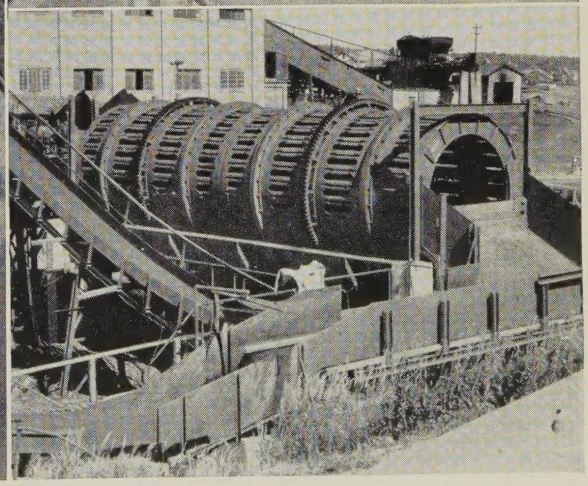

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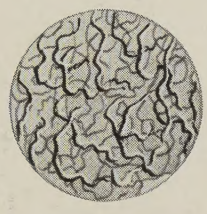

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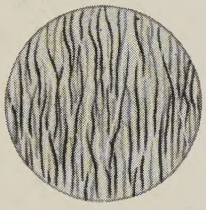

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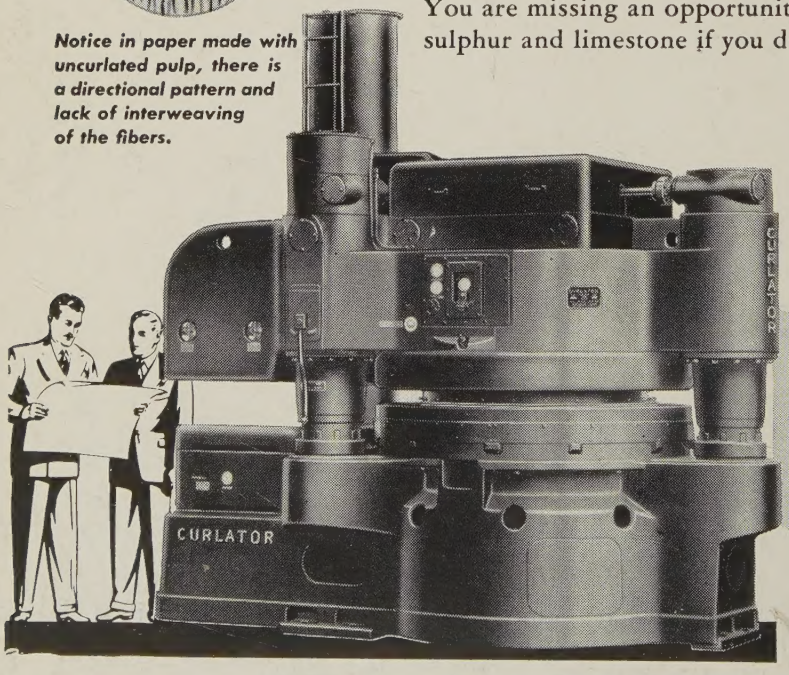

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